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We, the undersigned, hereby certify that we have
read the within thesis, entitled "AN INVESTIGATION OF THE
CHEMICAL AND PHYSICAL PROPERTIES OF THE ASH OF REPRESENTATIVE
ALBERTA COALS," submitted by Mr. A. G. Scroggie, B.Sc., in
fulfilment of the requirements for the degree of M.Sc. in
Arts & Sciences, and we hereby recommend its acceptance.

Edmonton, Alberta,

April 25, 1924

Thesis
1924
#7

AN INVESTIGATION
OF
THE CHEMICAL AND PHYSICAL PROPERTIES
OF THE ASH
OF REPRESENTATIVE ALBERTA COALS

By

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University of Alberta,

Edmonton, Alberta,

1923 --- 1924

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Introduction

The work described in this thesis was carried out at the University of Alberta under the auspices of the Scientific & Industrial Research Council of Alberta, and under the immediate direction of Mr. Edgar Stansfield, the Research Engineer on Fuels.

The Council provided in their laboratories the special furnace for the fusing tests, and fitted it up with all the necessary accessories.

The chemical analysis was carried out in the Industrial Laboratories of the University of Alberta, and the writer is deeply indebted to Mr. J. A. Kelso, the Director, for the facilities in the way of apparatus and chemicals that were placed at his disposal.

A number of boiler trials were observed, and these were for the most part run in the Power Plant of the University of Alberta. A few others that are referred to were run at Messrs. Trudeau's Dye Works, under the general direction of Mr. C. A. Robb, of the University Staff.

Acknowledgments

The writer wishes to acknowledge his indebtedness to many members of the staff of the Research Council and of the University, in particular,-

Mr. Stansfield, Dr. Lehmann, Mr. Kelso, Dr. Allan, Dr. Clark, Mr. Cameron, Mr. Pitcher, Mr. McMillan, Mr. Sanderson, Mr. Hollies, Mr. Campbell, and the other members of the staff of the Research Council.

AN INVESTIGATION OF THE CHEMICAL
AND PHYSICAL PROPERTIES OF THE ASH
OF REPRESENTATIVE ALBERTA COALS

CHAPTER I.- OUTLINE OF COAL TESTING

Historical

The history of man's first use of coal is lost in the mists of prehistoric time, but we know that at an early date its wonderful properties were discovered and put to use. It has been said that modern civilization began with the invention of the steam engine. At least it is an admitted fact that this great agency, which man put to so many uses, created a great demand for coal, and gave at the same time a means of supplying that demand; for by its use alone was the raising of coal and water from a depth made possible. Following its development, the production of coal became a great industry, coal itself a most important article of international trade, and those countries which had large reserves of this commodity automatically took a leading place in the economic and industrial world.

As time went on, the buyers of coal learned by experience that there was a great difference in coal from different places, and in recent years methods of testing coal have been developed

so that the intelligent buyer may obtain a good idea of what he is getting. Here in Alberta to-day we have great stores of coal of different kinds, and it is essential, if we are to realize the prestige and benefits that they can confer on us, that we thoroughly test our coals and establish their good properties.

The Scope Of This Thesis

As a part of this plan, the work described below is an application to representative Alberta coals of one of the more recently developed of these tests, viz., THE DETERMINATION OF THE SOFTENING TEMPERATURE OF COAL ASH. An endeavor is made to establish a relation between the theoretical results obtained in the laboratory, and the formation of clinker when the coals are burned in practice. A discussion of the general problem of clinker formation is included, and also of the improvement in the clinker trouble likely to be effected by washing the coal. The chemical analyses of typical samples of ash are given.

Earlier tests fall under three main headings:- Ultimate Analysis, Heating Value, and Proximate Analysis.

The Ultimate Analysis

This was an outgrowth of the development of modern chemistry,

and consisted in resolving the combustible portion of the coal into its elements, consisting mainly of carbon, hydrogen, and oxygen, with smaller amounts of sulfur and nitrogen. This test is not much used by present day purchasers.

The Estimation Of The Heating Value

This very important test determines the heating value of the coal in terms of B.t.u's, which are defined as the amount of heat required to raise one pound of water through one degree Fahrenheit. Roughly speaking, this test is carried on by igniting a small amount of coal in an atmosphere of oxygen, and measuring the amount of heat given off. The figure for Alberta coals varies over wide limits from 7,000 to 14,000 B.t.u.'s per pound on the basis of the coal as received. The general rule can be stated: that, of coals which can be obtained at the same price per ton, one should purchase that which has the greatest heating value -- or, in other words, the higher B.t.u.'s.

The Proximate Analysis

This test is applied to all coals; and in it the coal is divided into four arbitrarily defined parts, on a percentage basis, known respectively as:-

- (a) The Moisture Content, or water present in the coal, obtained by drying the coal for 1 hour at 105°C.
- (b) The Ash Content, or matter left after combustion

of the coal;

- (c) The Volatile Matter, obtained by heating the coal at about 950°C. for 7 minutes, with a provision for preliminary heating of high moisture coals: the loss in weight, less the moisture, being termed the volatile matter;
- (d) The Fixed Carbon, being the difference between the sum of the other three and 100.

The figures for Alberta coals again vary over wide limits:

The moisture from	1%	to	30%
The volatile matter from	10%	to	35%
The ash from	2%	to	40%
The fixed carbon from	40%	to	85%.

In the process of combustion, all but the ash pass off as gases, but the ash has to be disposed of mechanically; so that, generally speaking, it was considered wise, given a choice of coals at the same price per heat unit (such as, 30 cents per 1,000,000 B.t.u.'s) to choose the one with the least ash content.

The latter choice is, however, modified by the fact that in the furnace the ash sometimes melts and forms a hard, solid mass, termed clinker. The tendency to do this bears no relation to the amount of ash in the coal (See Figure 3.), but is an inherent property of the individual coal, and related mostly

to its chemical composition, but also dependent on other factors.

These other factors, as listed by Mr. E. B. Ricketts, chairman of the Sub-committee on Fusibility of Coal Ash Co-ordination of the American Society for Testing Materials, are:-

- (1) The caking qualities;
- (2) The manner in which the ash-forming material exists in the coal;
- (3) The manner in which it conglomerates;
- (4) The temperature of the fuel bed;
- (5) The time it is subject to this temperature;
- (6) The size of the particles;
- (7) The association of the particles.

This report was presented to the Society at their annual meeting in 1923, and shows that the problem has a great variety of factors, calling for much investigation and research.

CHAPTER II.- CLINKER AND ITS OCCURRENCE

Introduction

A broad division can be made between two main types of clinker: first, the large, porous masses known as "soft clinker", which give little trouble; and, second, the hard, solid mass formed by ash which has been heated till almost liquid and cools to a compact glassy mass which adheres to grates and cuts off the draft or causes trouble with the refractories. The effect of this clinker differs with the methods of stoking, and these will be considered separately.

Hand - Fired Furnaces

In a furnace of this type, the clinker is allowed to gradually accumulate on the grate till the depth of the refuse interferes with the draft. Owing to the grates being covered with clinker, unfused ash is unable to fall through, and the major portion of the ash has to be raked out by hand, involving a large amount of labor for the fireman. While this operation is being carried out, the doors of the furnace are open, and the cooling due to this, results in a drop in steam pressure, the loss in the average efficiency from this cause being as high as 5 to 10%. Cases are on record where coals giving bad clinker trouble have had to be cleaned every other hour.

(Blizard: Bulletin 27, Mines Branch, Ottawa, No. 76, page 39.)



PLATE I.- HARD AND SOFT CLINKER FORMATIONS

With coking coals it is hard for the fireman to distinguish between coke and clinker, with the result that an amount of the former is raked out with the clinker and is lost as fuel. On refiring there is also a loss of fuel, due to fines falling through the grate, and in some cases low volatile coal will not pick up quickly after the cleaning. Despite these disadvantages, however, this type of firing is practically a necessity with coking coals, which must be sliced at intervals to break up the coke. Lignite coals, on the other hand, do not coke, and therefore do not need to be sliced. The practice, indeed, results in heavy clinker formation. This is due in part to the facts that:-

Mixing the ash particles tends to lower the fusion point;

The fused ash, already in ideal proportions, is brought into the hottest part of the fire, where it readily liquefies and absorbs unfused ash;

Its place on the grate is taken by other clinker, so that there is a layer on the grates and more in the fuel bed;

A system of shaking grates reduces the trouble by causing the finer ash to fall through, so lessening the ash above the grates; but this will not work economically where there is much fine coal being fired. Spraying water or steam on the grates, or keeping water in the ash pit tends to lessen the amount of clinker, owing to the cooling effect on the grates and also in part to the fact that the water gas produced is burned in the

combustion chamber above the coal, the reaction that forms the gas absorbing heat from the coals; both of which factors tend to lower the temperature of the fuel bed.

An analysis of the figures in Bulletin 27 of the Mines Branch, Ottawa, quoted above, bears out the statements with regard to loss of combustible matter and to the proportion of the ash above the grates when using a hand-fired furnace under different conditions.

Overhead Stokers

The above remarks apply also to those types which have stationary grates and which require the removal of the ash by hand.

Chain Grate Stokers

In this form of stoker the clinker is, theoretically, carried over the end of the grate and dumped with the continuous revolution of the grate, thus doing away with the operation of cleaning the fire that is essential to hand-fired furnaces. In both types, however, the clinker sometimes sticks to the refractories; and in the chain grate stoker this is a serious matter, for the mass attached to the side wall bunches the coal in the middle of the grate, where it becomes so thick that it is not all burned and a portion is carried

over into the dump. In addition, on that part of the grate behind the clinker, the coal having been swept off, the air rushes through, and cools the tubes, causing a drop in steam pressure. The masses attached to the wall are often so securely set that they have to be knocked off with a hammer, and they often drag a portion of a brick out with them. This action on the refractories is probably the most serious of the effects of clinker formation, and often necessitates the rebuilding of a furnace wall in the course of a few years.

Methods of lessening this trouble with the refractories include:-

1. The thickening of the fuel bed at the side of the grate. This method is in use with considerable success at the University of Alberta Power Plant.
2. The building of a false wall of either hollow tile or brick along the edge of the chain grate. The wall is kept cool by a circulation of air, which, however, may cool the furnace if too much is allowed to flow into the combustion chamber, or, if allowed to pass out, occasions a distinct loss of heat.
3. Another method is to run a water tube along the wall, just along the side of the grate, but such a tube is liable to suffer from excessive scale trouble.

All of these methods are based on the theory of cooling the wall, and so preventing it from softening sufficiently for the clinker to stick to it.

4. An entirely different method of handling the trouble is based on the fact that the edges of the brick soften first; and it endeavors to solve the problem by using large bricks, three feet or more in length, thus lessening the liability of union between clinker and soft brick. Much the same effect can be achieved by lining the furnace with a layer of cement, but this is soon scaled off by removal of adhering clinker so that the bricks are exposed after a comparatively short time.

Underfeed Stockers

In the Jones type the chief difficulty with clinker is the cutting off of the draft by the liquid clinker flowing into the orifices. Moving grates are not used. Other under-feed types, such as the Taylor, have some difficulty with clinker adhering to the side walls as well.

Powdered Coal

This method of burning coal has recently been developed to a large extent. The coal is powdered and blown into the fire box by an air stream. This condition of fine division and intimate mixing is an ideal one for a low fusion temperature when there is an excess of the more readily fusible material, as is likely to be the case with low ash coals, but not in coals carrying a high content of siliceous material. (See below,

particularly Cadomin ash, under "Constitution of the Ash.") As might be expected from this type of coal, considerable difficulty has been encountered. In some cases a solid mass of clinker formed on the tubes, cutting down the transference of heat; and also at first much trouble was caused by the clinker forming on the bottom of the fire box in a solid mass that could not be removed. This difficulty has been overcome to a large extent by the introduction of the so-called water screen, which is really a series of water tubes occupying the space normally occupied by the grate. They are in connection with the boiler, and serve to cool the falling ash so that it is no longer liquid when it reaches the bottom. (See Savage, H.D.: Engineering Journal of Canada, June, 1923.)

General Considerations

Clinker trouble is aggravated as a general rule by running at high ratings or under any other circumstances which tend to raise the temperature of the fuel bed.

With good draft control most of the ash is normally cooled before it becomes liquid and can choke up the grates. If, however, anything interferes with this condition, such as too thick a fuel bed, leaky walls, or poor draft in the flue, the ash may not be cooled sufficiently to prevent the formation of much hard clinker on the grates; and as this clinker cuts the draft in its turn, the effect is an accumulative one.

Bone, in "Coal and Its Scientific Uses", page 47, says:
"It is commonly agreed that salty coals produce a far more rapid corrosion of the refractory oven walls than those that are comparatively free from salty constituents." This effect is much the same in the combustion chamber. Such salty constituents may come from the coal, or from a water it is washed with, or from the water sprayed on it before feeding, this being a common practice to keep slack coal together till it is ignited. The effect of these salty coals increases with the amount of moisture present, which in some way aids in the transference of alkaline elements to the refractories, where they form a glaze which is brittle and easily broken by the wear of the clinker, the wall being thus gradually eaten away.

The use of slack coal as a rule results in the formation of more clinker than lump coal from the same mine. Primarily, this is due to the fact that slack generally has a higher ash content, but other factors also enter in. When slack is used, the same rate of firing results in a thinner and hotter fire, and the higher temperature will result in more clinker. The slack packs closer together, also, and if fed too thickly will choke the draft to some extent, and may prevent a measure of the cooling action referred to above. Sinatt tested the softening temperature of the ash from different commercial grades of the same coal, and found that, the finer the coal, the lower the softening temperature, was the rule. He gives figures

showing the softening temperatures of lump coal, slack coal, and slurry from the same mine; and in the three cases quoted there was a decrease, in the order given, of the softening temperature. He mentions that the slack and slurry were washed, but does not discuss the bearing of this on the softening temperature, and it will be shown below that this is sometimes quite significant. When the softening temperature of the ash is lower in the fine coal, it will be due, most likely, to the better mixing of the ash constituents if there is an excess of low fusing components. (See above, "Powdered Coal", and below, "Constitution of the Ash"; Sinatt: Coll.Guard., Jul.'23,p.21.)

There would appear to be a widespread opinion among firemen that the use of damp or wet coal increases the tendency to clinker. This has not been verified, and if wet coal has this effect, no satisfactory explanation has been found. Attention might be drawn, in this connection, to the action of the water sprayed on the grates, as mentioned above, by which the tendency to clinker is lessened; and to the part played by the moisture content with salty coals, mentioned above. This would suggest the use of steam for dampening the coal. There is also the possibility that the moisture in the coal causes it to break up more rapidly, exposing fresh surfaces to combustion very quickly, and setting the volatile matter free sooner than it is given up by dry coal. These factors would increase the temperature of the fire, and so the amount of clinker.

These are some of the manifestations of the clinker problem which have caused much investigation and experimentation, leading finally to the Standard Method for the determination of the softening temperature of coal ash. An historical outline of the more important of these researches is given immediately below.

CHAPTER III.- THE DETERMINATION OF THE
SOFTENING TEMPERATURE OF COAL ASH.

H i s t o r i c a l

In 1895 and 1897 Prost tested a number of Belgian coals, placing a few grams in a porcelain crucible, and heating the ash to different temperatures estimated by means of gold and platinum alloys. He obtained different temperatures with different gases, and was not successful in relating the softening temperature to the chemical composition of the ash.

In 1902 Chatelier introduced the Seger cone comparison method for estimation of the fusing point of coal ash. He made his cones with an organic binder, and took as the softening point the temperature when they had become half their initial height.

In 1904 Cobb used small pyramids, and a pyrometer for measuring the temperature. He called attention to the fact that a regular rate of heating was essential to check results.

In 1910 Bailey and Calkins, and also Hitchcock, after estimating the softening temperature of two coals, fired them in a hand fired Dutch oven. They found a general agreement between softening temperature and clinkering properties. Bailey concludes that "the main factor in the formation of clinker is the difference between the fusing temperature of the ash and the temperature to which it is subjected. Any factor such as

excess air, rate of combustion, and thickness of fuel bed, which will affect the temperature, may cause a corresponding change in the amount and nature of the clinker from a given coal."

In 1910 Stansfield, working on Canadian coals, used cylinders of ash; but he could not see them at high temperatures, and so adopted the method of heating separate cylinders of the same ash to different temperatures as determined with a Morse optical pyrometer. He found it difficult to see much connection between his results and the data taken on clinker formation at boiler trials on the same coals.

In 1910 Marks, using a cone method, mixed various foreign substances with the same ash. He found that aluminium oxide softening raised the temperature of the ash; while other substances, including CaO, Fe_2O_3 , Fe, FeS_2 , CaS_2 , $CaSO_4$, lowered the same of heating. Of these latter, iron sulphide had the most effect.

In 1913 Constan, working with 200 different European coals, measured the temperature of initial deformation of the cone, and the temperature of fluid point, with an optical pyrometer. He concluded that:-

The proportion of ash does not influence the softening temperature unless slate or pyrite are present.

The softening temperature is characteristic of the seam.

The softening temperature ranges from 1150 to 1700°C.

In 1914 L. S. Marks called attention to differences of as much as 390° obtained for the same ash in different laboratories. He himself obtained more than 250° difference on the same sample in two different types of furnace. He finally adopted a method of mounting cones in a horizontal position, heating them at the rate of 2° a minute, measuring the temperature with a thermo-couple, and maintaining an oxidizing atmosphere. Comparing his results with actual clinker formation, he concludes that a general relation was shown between the two, but not definite enough to be reliable.

In 1914 Hubbley brought out a fusiometer which recorded the shrinking of a cone, showing its relative viscosity and final collapse on a scale. The method was fairly successful, but depended more or less on its particular set-up, and could not necessarily be duplicated in other laboratories.

In 1915 Palmenberg mounted cones in the middle of a muffle of a meker furnace, and took as the softening temperature the time at which the cone had bent till it touched the base.

In 1915 Ricketts mounted the cones horizontally in a fired hessian crucible in a gas/melting furnace. He observed the cones by means of a quartz tube through the side of the furnace and the crucible. The temperature of fusion was taken when the cone reached a 45° position. This method gave uniformly good results.

Cameron of Saskatoon did some work on Alberta coals, but his results are not available.

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Development Of The Standard Method

Because of the importance of the clinkering problem, as pictured above, and because different workers got such widely differing results when testing the same coal in an endeavor to determine its clinkering tendency, the Bureau of Mines, Washington, was induced to study the question and to try to develop a method for testing coal that would give check results and that would give a comparative measure of the softening temperature of the particular ash being tested. The work was carried out at the Pittsburg Station of the Bureau of Mines, under the direction of Mr. Fieldner. Some of the experiments are reported in the Journal of Industrial & Engineering Chemistry for 1915, pp. 399-474, and another description of the work is given in Bulletin No. 129 of the Bureau of Mines, Washington.

They conducted a number of experiments with a variety of furnaces, including:-

1. Platinum wire resistance furnace, with atmosphere of air;
2. Meker muffle furnace, with atmosphere of air and combustion gases;
3. Muffle No.2, with atmosphere of air and combustion gases;
4. Molybdenum wire resistance furnace, atmosphere H_2 or $H_2 & H_2O$;
5. Granular carbon resistance furnace, atmosphere CO;
6. Northrup graphite resistance furnace, atmosphere CO.

They found that they could get the best checks in an atmosphere of air or in a strongly reducing atmosphere, as in

the carbon resistance furnaces, but that the results were uniformly higher than in the moderately reducing atmospheres of the muffle types. Results varied between the types by a large amount, being roughly from 130 to 325°C. This was traced to the iron content of the ash, and it was found that the lowest results were obtained in the furnaces where the iron was reduced to the ferrous state, while in the other furnaces, where the iron was reduced to the metallic state or left in the ferric condition, results were uniformly higher. The method of preparation of the ash, the shape, size, and inclination of the cones, the rate of heating, and the effect of different reducing atmospheres, such as mixtures of CO and CO₂, and of H₂ and water vapor, were all studied.

From these experiments and others that showed that the iron in clinkers was principally in the ferrous state, together with the knowledge that the greater portion of the fuel bed was subject to a reducing atmosphere, they concluded that such a reducing atmosphere was essential to secure results comparable with actual furnace conditions. It was found that the desired degree of reduction was obtained with from 30% to 70% of reducing gases present, and that this ratio was produced by an atmosphere of combustion gases such as were found in a muffle furnace. Other conclusions were: that the cones should be mounted vertically, and should not be slender, as they would then have a tendency to premature bending because they were weak structurally; and that they should be of a size that would

fuse to a ball rather than bend over; and that a variation between 5 and 10° per minute in the rate of heating made no appreciable difference in the softening temperature. (Bulletin 129, U.S. Bureau of Mines, Washington.)

The Standard Method -- A. S. T. M. D-22-23.

These results were embodied in a tentative method that was later adopted as standard by the American Society for Testing Materials in 1923. This is briefly as follows:-

The sample of powdered coal is ignited in fireclay dishes in an electric muffle furnace till completely oxidized. The temperature is kept at about 800 to 850°C. to prevent premature fusion. The ash is then ground in a silica mortar till it passes a 200-mesh sieve, and is then ignited in a current of oxygen for two hours to insure complete and uniform oxidation. The ash, of which about 10 grams is required, is moistened with 10% dextrin solution, and molded into cones formed as three-sided pyramids, 3/4-ins. high, and 1/4-in. along the sides at the base. This is done by pressing the plastic ash into a brass mold of the dimensions given. The cones are then mounted on an alundum or other refractory base, and heated to dull red heat for a half-hour to remove all carbonaceous material.

The base and cones are then placed in a specially fitted no.3 Melter's furnace. (See Fig. I. and Plate I., page 23.) The fittings consist of two nested crucibles: C, the inner one,

and B, the outer, which acts as a muffle. The larger one has two holes; one, E, for observation; and the other, in the same plane and at right angles to it, for a pyrometer tube and a blowing tube, F. The holes in the crucible are matched by similar holes in the wall of the furnace, E and I. This type of furnace is fitted with three tangential burners, which heat the crucible very uniformly, and at high heats the cones, D, mounted on the base, H, cannot be seen. They are brought into view by a momentary blast through the tube F, connected with the air supply. A reducing atmosphere is maintained by burning an excess of gas over air, so that a flame at least six inches high is obtained above the furnace. The rate of heating above 800°C. is between 5 and 10°C. per minute. The cones are viewed by means of a silica observation tube cemented into the furnace wall and fitted with a thin glass cover. The temperature is measured by a thermocouple or optical pyrometer. Readings are made every other minute, and the points noted:-

1. Where there is the first perceptible bending of the cone.

This is called the "temperature of initial deformation."

2. Where the cone has fused down to a spherical ball, or where it has bent to touch the base. This is called the "softening temperature."

3. Where the cone has become fluid and spread out on the base.

This is called the "fluid temperature."

The range of temperature between the first and second points is

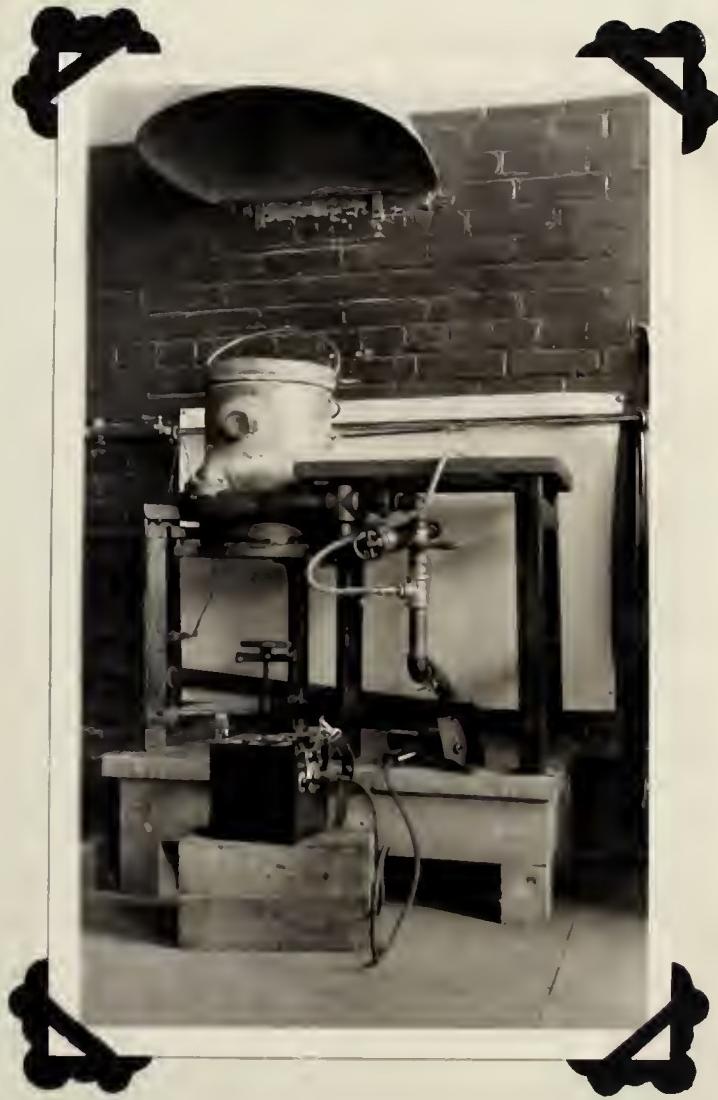
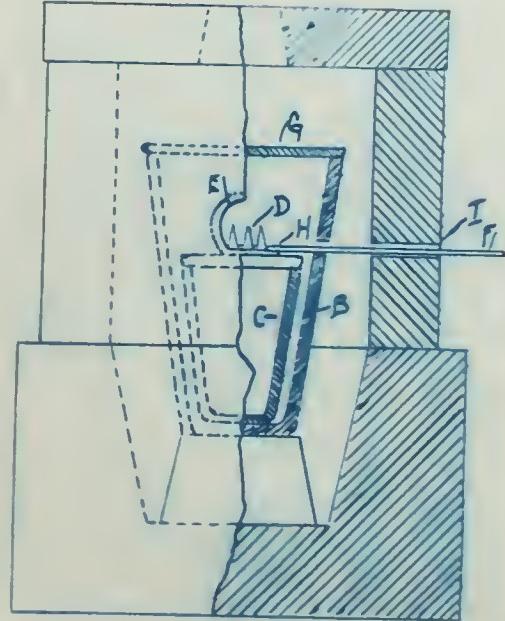


PLATE II.- FURNACE USED IN STANDARD METHOD FOR DETERMINATION OF THE SOFTENING TEMPERATURE OF COAL ASH.

FIGURE I.



SECTION OF NO. 3 MELTERS FURNACE
ARRANGED FOR FUSION TESTS
ASTM-^{from} STANDARDS



known as the "softening interval", and that between the second and third points is called the "fluid interval." The intervals are important as a measure of the comparative viscosity of the melting cone.

Comments On The Standard Method

One or two slight variations in the procedure were introduced. In the initial heating, silica dishes were substituted for fireclay, since these frequently broke, and the sample had to be discarded owing to the introduction of foreign matter. The silica dishes had a very much longer life. Moreover, when a particle came off the silica dish, it was generally in the form of a flake and easily detected, or if so small as to be missed it was an addition to the constituent already present in the greatest proportion and so caused the least possible percentage error. In both kinds of dish there was a deposit of ferruginous material, but in the silica dishes this appeared to be smaller although they were in use longer.

Ignition for two hours in oxygen did not seem to be sufficient time, judging by the mottled color effects that were produced. To prolong the time or to use less ash in a dish meant the use of more oxygen, but the same result was achieved by giving the ash a long preheating in the muffle before the oxygen was turned on.

Silica and Impervalite porcelain tubes were used for the

air blast, the former having a much longer life than the porcelain, though this was not long in either case. The silica was difficult to pierce. The method finally used was to grind more than half-way through the tube with the edge of an emery wheel, and finish with the edge of a new triangular file. The impervialite tubes were readily prepared with a file. The life of both tubes was prolonged by putting around them a band of fireclay or alundum cement at the place where they were exposed to the direct flame. This served to keep them from bending at the high temperatures. Bending caused a displacement of the air blast, and, when pronounced, the tube sometimes fused with the large inside crucible. Subsequent cooling, or jarring of the crucible in opening it after a run, would often break the tube.

It was found very beneficial to cover the top of the inner crucible with pure granular alundum. This neither fused nor caked at the temperatures reached, and neither the tubes nor the cone bases stuck to this inner cover, which formed the floor of the muffle. Bone ash and pure calcium oxide were tried, but neither were successful at the high temperatures.

It was also found essential to use some means of absorbing the vibration set up by the fan and motor, or else it shook the furnace. This was accomplished by the introduction of a piece of heavy hose in the air line. Previously the vibration had had the effect of tipping over unsteady cones, and of breaking the blowing tube.

Another precaution necessary was to obtain an air-tight

seal around the cover glass. Two methods were tried: asbestos packing, followed by a clamp and cement; or straight cement holding the brass sleeve to the silica tube. The latter was simpler, and seemed to give just as satisfactory results.

Accuracy Of The Method

The figure reported is in every case the mean of at least two runs in the furnace, and from three to five cones were used for each sample. Check readings on the point of initial deformation and on the fluid temperature were sometimes hard to get, because the first readings were lowered by warping of the cone, or by deformation due to structural defects, or warping following a strong blast from the air tube. The same readings might be raised by cones shrinking in on themselves without bending at all. In this case the point where the shrinking was first definitely noticed was taken as the point of initial deformation.

In the case of the fluid temperature, cones would sometimes go out of sight, but an examination of the plate after the furnace had cooled would reveal that they had been barely liquid. Since two kinds of cones were mounted at once to save time, this check was only available on the highest melting cones unless both varieties were fluid at the same temperature. When the difference was very great, one set of residues was often volatilized.

In the case of softening temperatures, exact checks were often obtained, and the range rarely exceeded 10°C.. It is probable that the limit of error on this reading was not more than 10°C.. The A.S.T.M. specifications allow a tolerance of 30° for the same operator, and the results are certainly well within this limit.

The runs were all conducted with the same relative excess of gas over air to produce the necessary reducing atmosphere; and the same kind of gas, Viking natural gas, was used for every determination.

In all the tests the same optical pyrometer was used. It was of the Leeds & Northrup type, the measurement depending on the matching of the color of a loop of wire, heated to incandescence by a measured current, with the color of the body whose temperature was to be measured. The instrument has a range of 500°C. on its scale, but this is extended by the use of a color filter which absorbs some of the rays, giving lower readings, and so enabling higher temperatures to be estimated. In use, the lower range runs from 800°C. to 1350°C., and the higher range from 1250°C. to 1700°C. In practice, the change was always made at 1300°C.. In this particular instrument there was a lag of about 25° between the ranges. It was checked by estimating the fusing temperature of pure strips of copper and nickel wire. The copper melts at 1083°C., and the average reading was 1085°C. The nickel melts at 1452°C., and the

average readings were about 25° low. Considering the lag above mentioned between the two ranges, and the lag on the melting point of pure nickel, all temperatures estimated on the high range were given a plus correction of 25°.

This method of testing by means of copper and nickel is recommended by the Bureau in Bulletin 129, but no specifications as to the method are given. It is justified, because in the furnace a reducing atmosphere is maintained, and very little oxide of the metal is formed. In these tests pieces of wire of about 26-gauge were used.

Readings on the instrument were always taken in the same way, and in all cases only those were accepted which checked within 2° on the scale. This corresponds to from 3 to 7°C., depending on the temperature.

Other Recent Methods

At least two other furnace designs have been described since the A.S.T.M. published its tentative standard.

A micropyrometer furnace was described by Fieldner, Selvig and Parker (J.Ind.& Eng.Chem., June, 1922, p.695.) This method consists of fusing a small amount of ash on a strip of platinum which serves as a heating element. The platinum is enclosed in a small alundum furnace mounted on the stage of a microscope which has an attachment fitted with a tungsten filament. The operator watches the progress of fusion of the ash through the

microscope, and the filament is used to estimate the temperature in the manner of an optical pyrometer, the filament being matched with a spot on the platinum strip beside the fusing ash. An oxidizing atmosphere is normally present, or the gases from a muffle furnace can be aspirated through the alundum furnace to get a reducing atmosphere similar to that of the standard method.

Advantages claimed are the ease and quickness of the method, and the comfort of the operator.

Compared to the results obtained with the standard method, the micropyrometer furnace gives a wide difference in an oxidizing atmosphere. With a reducing atmosphere, however, for ash of medium fusibility, the results are quite comparable. Ashes of low fusibility gave higher results, and those of high fusibility gave lower results. No definite conclusions were reached.

Another type of furnace was described by Mr. Sinatt, of the Lancashire & Cheshire Coal Research Board, in a paper delivered before the Manchester section of the Society of Chemical Industry (abstracted in Colliery Guardian, July, 1923, p.21.). He uses a silica tube as a muffle furnace, and heats the tube by means of an oxyhydrogen blowpipe. The ash is pressed into small cylinders, and mounted on a strip of platinum foil which is cleaned with hydrofluoric acid. The temperature is measured with a thermocouple, but no mention is made of any difficulty in seeing the cylinders. No provision is made for a reducing atmosphere, which would seem to be a grave defect. Fusions of standard cones quoted in the paper also gave wide variations from normal. Presumably some

modification could be made to insure a reducing atmosphere similar to the one described by Fieldner above, or by a mixture of hydrogen and water vapor; but possibly these would cut off the view of the cylinder. Advantages are the small amount of ash necessary, and the cheapness of the furnace. No comparisons with the standard American method are given, though one was obtained in this work. (See below, "Constitution of the Ash.)

CHAPTER IV.- SOURCE OF SAMPLES INVESTIGATED

The ash of a coal is the matter left after complete ignition; and is to be distinguished from the mineral matter originally in the coal, as certain constituents such as chlorides, carbonates, and sulphur, are more or less completely volatilized when the coal is burned. The ash comes from three distinct sources, which may be divided as follows.

1. The intrinsic ash, which is formed by the mineral matter derived from the original coal-forming plants, together with external matter intimately mixed with the deposits during their formation.
2. The extraneous ash, due to impurities laid down as partings, veins, and nodules, either at the time of deposition or by infiltration at later dates. These are composed mostly of clay, shale, slate, pyrite, or calcite.
3. The third source of ash is the mechanical mixture of fragments of the roof and floor due to the method of mining.

Ash from sources 2 and 3 can often be reduced by a process of washing, but this has little or no effect on the amount of intrinsic ash. Fieldner (Bulletin 129, U.S.Bureau of Mines, p.14.) says: "Such extraneous impurities as sandstone or shale tend to raise the softening temperature. Limestone and pyrite tend to lower it, although limestone, if added in excess, will raise the softening temperature."

In Alberta, pyrite is only present in small amounts, and

most of the coal beds lie between strata of sandstone or shale, so that low-ash coals should presumably have lower softening temperatures than those with high ash. Clinker formation, too, is apparently due in a large degree to the amount of low-fusing constituents that the ash contains. (See "Constitution of Ash", below.) For these reasons, therefore, it is considered that a sample from coal with little ash will give a better indication of its potential clinkering properties than a commercial sample with a greater amount of extraneous matter included.

The securing of representative samples, the first part of the work, was undertaken by the Scientific & Industrial Research Council of Alberta. Most of them were taken from the larger samples sent in by the provincial mine inspectors, who personally take the samples in the mines. In most cases the remarks accompanying these samples point out that they are quite representative of the coal as shipped, with the one exception, that no matter from the roof or floor is introduced. A few of the samples were obtained from the commercial samples supplied to the Council at different times, and these are possibly more representative, though this is open to question.

The general plan followed in this investigation was to examine at least two samples from each of the more important producing areas, and one from the smaller ones, where possible. Thirty-three samples in all were taken from the different parts of the province. They are divided as follows:-

Bituminous samples..... 7, numbered 1 to 7;

Sub-bituminous samples... 5, numbered 11 to 15;

Lignite samples..... 21, numbered 21 to 41.

Table No.1 gives a list of the different samples, and indicates their geological,geographical, and commercial sources. The origin of the samples, whether obtained commercially or through the mine inspectors, is indicated, as well as the serial number used in the investigation. Figure 2 indicates the geographical distribution of the samples on a map of Alberta.

The map of Alberta coal areas, recently compiled by Dr. Allan, shows 36 divisions, while only 20 were sampled. Owing, however, to the fact that some areas produce little or nothing, the samples taken are quite representative of the province. This is brought out by an analysis of the tonnage reports, as published by the Mines Branch, Alberta, for 1922, the latest figures available. In that year the areas sampled produced 100% of the bituminous coal, 99% of the sub-bituminous coal, and 95% of the lignite coal mined in the province. There was no anthracite being mined during the period when samples were being secured. These figures would indicate that the province was thoroughly sampled.

From these samples of coal, the ash was prepared as described above; and since it was thought that later work might be done on the ash, large amounts, from 30 to 50 grams of each sample, were prepared. Samples were taken of all the coals

on hand in the spring of 1923, and from all the areas available up to the first of March, 1924.

Samples were also prepared from a coal which had been washed, and from two coals that had been treated with heavy solutions.

TABLE I.- SOURCE OF SAMPLES

NO.	SERIES & Area	MINE	POST OFFICE	SOURCE	SEAM
	KOOTENAY	B I T U M I N O U S			
..	Smoky River.	No production
1	Brule.....	Blue Diamond Coal Co. ...	Brule	Ins.	#2
2	Mountain Pk.	Luscar Coal Co.	Luscar	Ins.
3	" "	Cadomin Coal Co.	Cadomin	Com.
4	Nordegg.....	Brazeau Collieries ...	Nordegg	Ins.	#2
..	Clearwater..	No production
..	Panther.....	No production
5	Cascade.....	Canmore Coal Co.	Canmore	Ins.	S & C
..	Highwood....	No production
..	Oldman.....	No production
6	Crowsnest...	Hillcrest Collieries ...	Hillcrest	Ins.	#1
7	"	Greenhill Mine	Blairmore	Ins.	#1
	BELLY RIVER	S U B - B I T U M I N O U S			
..	Halcourt....	No production
..	Prairie Ck.	No production
11	Coalspur....	Macleod River Hard Coal Co.	Mercoal	Ins.	#1
12	"	Foothills Collieries, ...	Foothills	Com.	#1
13	"	Sterling Collieries ...	Sterco	Ins.
14	Saunders....	Saunders Ck.& Big Horn Coll's	Saunders	Com.	#2
..	Morley.....
..	Pekisko.....
15	Pincher.....	Estell Mine	Lundbreck	Ins.	#1
		(Continued on next page)			

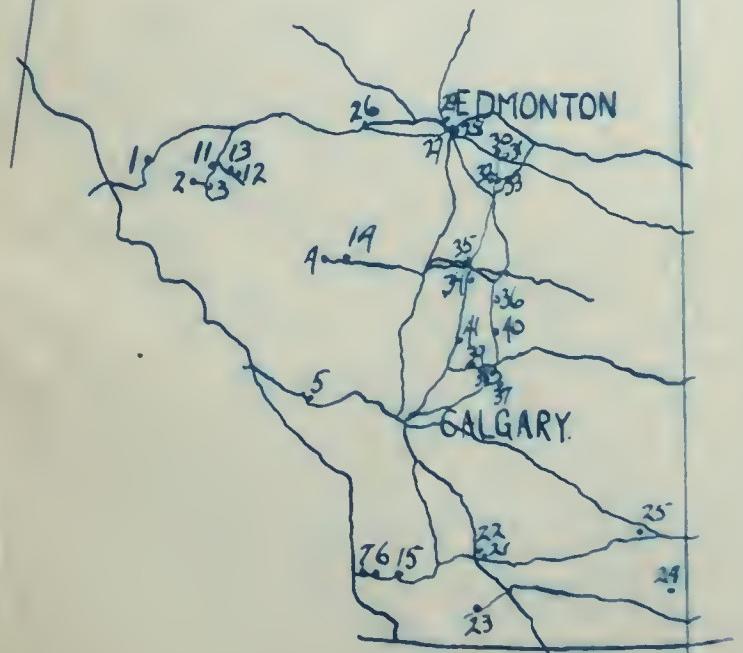
TABLE I.- SOURCE OF SAMPLES (continued)

NO.	SERIES & Area	MINE	POST OFFICE	SOURCE	SEAM
BELLY RIVER					
		LIG NITE			
21	Lethbridge..	Galt No.6	Lethbridge	Ins.	Leth.
22	"	North American Collieries	Coalhurst	Ins.	Leth.
23	Magrath.....	B. Welsh	Magrath	Ins.
..	Taber.....
..	Milk River..
24	Pakowki.....	Geo. Bradich	Thelma	Ins.
25	Redcliff.....	Redcliff Brick & Coal Co.	Redcliff	Ins.
..	Brooks.....
EDMONTON					
26	Pembina.....	North American Collieries	Evansburg	Ins.
27	Edmonton....	Penn Mine	Edmonton	Com.	Lower
28	"	Twin City Mine	Edmonton	Com.	Lower
29	"	North Star Coal Co. ...	Cardiff	Ins.	#1
30	Tofield.....	Tofield Coal Co.	Tofield	Ins.	Upper
31	"	Dobell Coal Co.	Tofield	Ins.	Upper
32	Camrose.....	Canadian Dinant Coal Co.	Dinant	Ins.	#1
33	"	Spicer Coal Co.	Dinant	Ins.	#1
34	Ardley.....	Mutual Mines	Ardley	Ins.	Upper
35	"	North Star Coal Co. ...	Alix	Ins.	Upper
..	Castor.....
36	Big Valley..	Big Valley Collieries ...	Big Valley	Com.	Upper
..	Sheerness...
37	Drumheller..	Rose Deer Coal Mining Co.	Wayne	Ins.	#1
38	"	Western Gem Mining Co.	Drumheller	Ins.	#5
39	Carbon.....	Peerless Carbon Coal Mines	Carbon	Ins.	Upper
40	"	Clyde Wooden Mine ...	Rumsey	Ins.	Upper
41	"	Ellis Coal Co.	Three Hills	Ins.	Upper
..	Gleichen....
..	Champion....

FIGURE 2.

S K E T C H - R A I L W A Y - M A P
O F T H E
P R O V I N C E O F A L B E R T A

ILLUSTRATING SOURCE OF ASH SAMPLES



CHAPTER V.- INVESTIGATION OF PHYSICAL PROPERTIES OF ASH SAMPLES

Bone, in his book, "Coal And Its Scientific Uses", says; "Variations may occur both in amount and character of the ash content along a particular seam. Such differences are usually gradual as long as they relate to the same horizon of the seam, but they may be abrupt in a vertical direction, especially when successive layers show marked differences in texture."

From this we may conclude that the physical properties, including the softening temperature of the ash, of samples from the same area would be very similar if the samples represent the same seam, providing the distance between the points on the seam is not too great, and that each sample represents the whole seam.

The results of the investigations into the physical properties of the ash samples, together with the conclusions drawn from them, have been divided for convenience into three groups, as follows:-

1. Conclusions depending on the COLOR of the ash;
2. Conclusions depending on the SOFTENING TEMPERATURE of the ash;
3. Conclusions depending on the CONSTITUTION of the ash.

These will now be considered in the order given.

Conclusions Depending On Color

The similarity in properties forecasted above was first

brought out in a distinctive manner by the color assumed by the ash, following its ignition in oxygen. The colors ranged through deep chocolate, buff-red, tan, yellow, cream, and grey.

In the areas from which duplicate samples were taken, the colors compared favorably, on the whole, as shown in Table II.

TABLE II.- DIFFERENCES IN COLOR OF ASH SAMPLES
FROM THE SAME AREAS

Penn & Twin City, similar; Cardiff, distinctly different.

Galt & Coalhurst, similar.

Alix & Ardley, similar.

Carbon & Three Hills, similar; Rumsey, distinctly different.

Mercoal & Foothills, similar; Sterling, distinctly different.

Greenhill & Hillcrest, similar.

Luscar & Cadomin, slightly different.

Wayne & Drumheller, distinctly different.

Dobell & Tofield, distinctly different.

Spicer & Dinant, distinctly different.

In only the last two cases is there a distinct difference in color recorded between samples that are from the same seam, while in most cases where the color is the same, the identity of the seam is fairly well established.

In the two cases just mentioned, the Tofield and Camrose areas, the samples show in each case a difference in color, though the mines are on the same seam. The ash content is approximately the same, and they are quite close geographically. There is no known fault between the mines in either case, but both coal beds

lie close to the surface. In the Tofield area the cover is only 15 feet, and at Camrose, 30 feet. It is possible that this strata has allowed a seepage of water into a portion of the coal seam, which might change the color of the ash, or that at some time part of this cover was eroded away, and the coal exposed to the weather or to iron-bearing waters. A suggestion of this latter idea is conveyed in the remarks of the sampling inspector, who notes in one case that in some parts of the mine the slips are colored yellow. This would appear to be worthy of further investigation, for it might be possible to use the color scheme for identifying seams.

The color of an ash is due, presumably, to its various constituents, of which the amount and kind are functions of the locality. A reddish color is given by the iron present, and a lighter color by the other ingredients, and it might be thought that since iron plays so important a part in the fusing of ash that the higher the color, the lower the softening temperature, would be the rule. This is true in general; but there are so many exceptions that color cannot be relied on as an index of the softening temperature. The only statement that can be made is that no red-colored ash was found that had a fusing temperature above $1250^{\circ}\text{C}.$, nor any light colored one which fused below $1100^{\circ}\text{C}.$.

The color could be used to detect the substitution of certain kinds of coal for others, in some cases. A list could

probably be drawn up, including most of the lignites and Saunders coal, which could not be substituted for other coals, including most of the bituminous and sub-bituminous. The color test could easily be made, following the usual ash determination of the coal, and would be simpler than running a determination of the heating value, though not so conclusive. In cases where samples were fraudulently dried before analysing, the color might be the more reliable test.

In general, high ash coals have a light colored ash, while coals with low ash contents give colored samples; so that it is possible that the intrinsic ash from all coals or from any one kind of coal would be the same in color, this color being most likely of a pink or reddish shade. Some work was done along this line in order to establish the uniformity of color, if it existed, and to see if intrinsic ash from different sources had the same softening temperature, but it was not completed in time for inclusion in this report.

Conclusions Depending On The Softening Temperature

In this connection, two tables are given, showing the results of the determinations of the softening temperature of the ash samples. Table III. gives the results in °F., and Table IV. gives the same results in °C.. From this latter table a graph has been prepared (Figure 3) in which the temperature of initial deformation, the softening temperature, and the fluid temperature

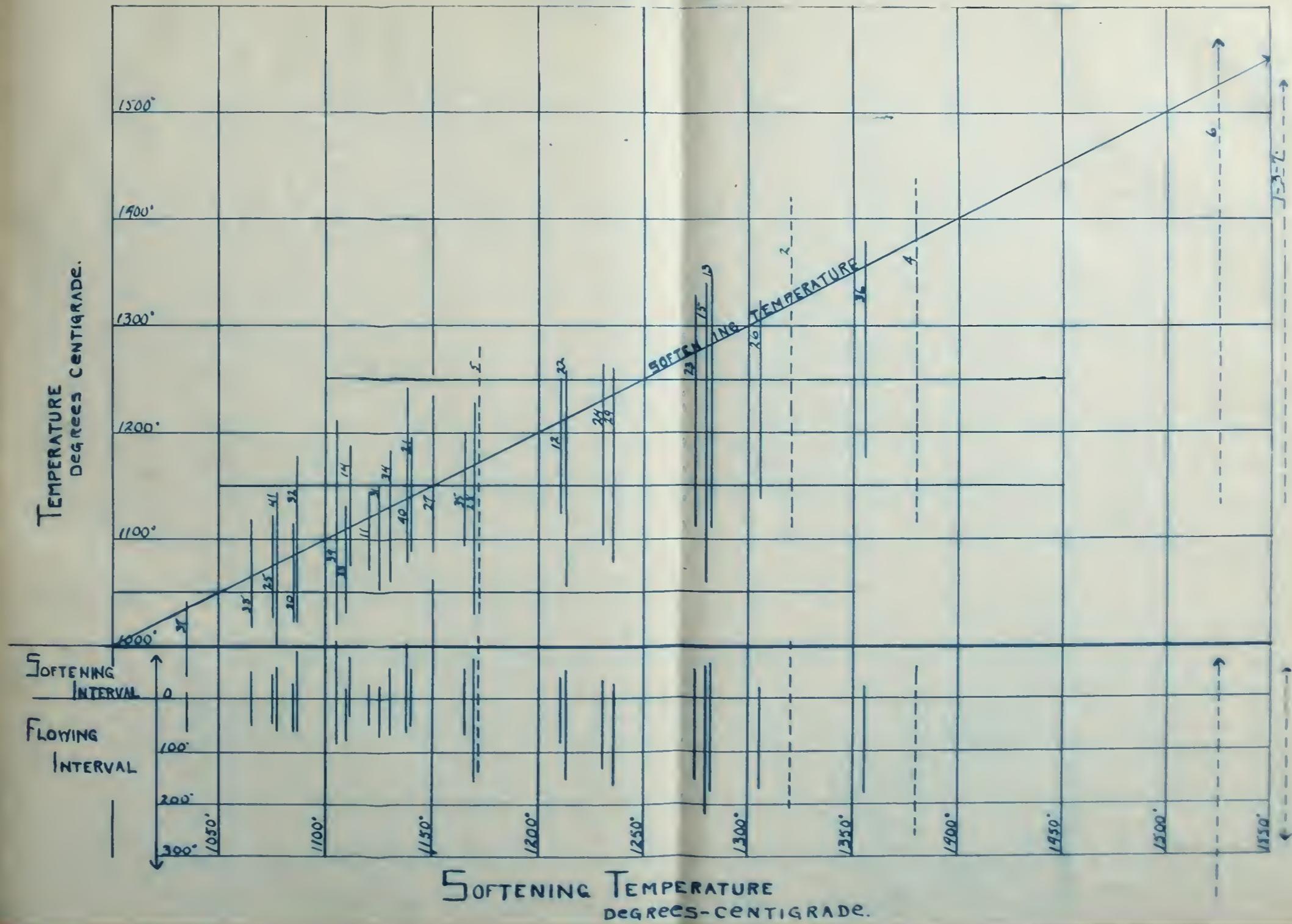
TABLE III.- FUSION DATA FOR SAMPLES, °F.

Area	No.	Mine	Softening Temp.	Softening Interval	Flowing Interval
BITUMINOUS					
Brule.....	1	Blue Diamond.....	2820	720	
Mountain PK.	2	Luscar.....	2410	375	180
" "	3	Cadomin.....	2820	765	
Nordegg....	4	Brazeau.....	2515	480	110
Cascade....	5	Canmore.....	2140	250	210
Crowsnest..	6	Hillcrest.....	2775	710	
	7	Greenhill.....	2820	755	
SUB-BITUMINOUS					
Coalspur...	11	Mercoal.....	2050	90	45
"	12	Foothills.....	2210	155	70
"	13	Sterling.....	2335	310	115
Saunders...	14	Saunders.....	2030	65	145
Pincher....	15	Lundbreck.....	2335	395	110
LIGNITE					
Lethbridge.	21	Galt.....	2085	90	100
"	22	Coalhurst.....	2210	280	100
Magrath....	23	Welsh.....	2325	280	100
Pakowki....	24	Bradich.....	2245	240	65
Redcliff...	25	Redcliff.....	1965	90	90
Pembina....	26	North American.....	2380	305	35
Edmonton...	27	Penn.....	2100	125	150
"	28	Twin City.....	2140	290	150
"	29	Cardiff.....	2255	290	45
Tofield....	30	Tofield.....	1985	115	45
"	31	Dobell.....	2060	135	35
Camrose....	32	Canadian Dinant.....	1985	115	160
"	33	Spicer.....	2030	145	35
Ardley.....	34	Ardley.....	2065	125	100
"	35	Alix.....	2130	125	100
Big Valley	36	Big Valley.....	2470	325	45
Drumheller	37	Wayne.....	1895	115	10
"	38	Drumheller.....	1950	90	95
Carbon.....	39	Carbon.....	2020	155	190
"	40	Rumsey.....	2080	110	180
"	41	Three Hills.....	1965	135	100

TABLE IV.- FUSION DATA FOR SAMPLES, °C.

Area	No.	Mine	Softening Temp.	Softening Interval	Flowing Interval
BITUMINOUS					
Brule.....	1	Blue Diamond ...	1550	400	
Mountain Pk.	2	Luscar ...	1320	210	100
" "	3	Cadomin ...	1550	425	
Nordegg....	4	Brazeau ...	1380	265	60
Cascade....	5	Canmore ...	1170	140	115
Crowsnest..	6	Hillcrest ...	1525	395	
"	7	Greenhill ...	1550	420	
SUB-BITUMINOUS					
Coalspur...	11	Mercoal ...	1120	50	25
"	12	Foothills ...	1210	85	40
"	13	Sterling ...	1280	170	65
Saunders...	14	Saunders ...	1110	35	80
Pincher....	15	Lundbreck ...	1280	220	60
LIGNITE					
Lethbridge.	21	Galt ...	1140	50	55
"	22	Coalhurst ...	1210	155	55
Magrath....	23	Welsh ...	1275	155	55
Pakowki....	24	Bradich ...	1230	135	35
Redcliff...	25	Redcliff ...	1075	50	50
Pembina....	26	North American ...	1305	168	20
Edmonton...	27	Penn ...	1150	70	85
"	28	Twin City ...	1170	160	85
"	29	Cardiff ...	1235	160	25
Tofield....	30	Tofield Coal ...	1085	65	25
"	31	Dobell ...	1125	75	20
Camrose....	32	Canadian Dinant	1085	65	90
"	33	Spicer ...	1110	80	20
Ardley.....	34	Ardley ...	1130	70	55
"	35	Alix ...	1165	70	55
Big Valley.	36	Big Valley ...	1355	180	25
Drumheller.	37	Wayne ...	1035	65	5
"	38	Drumheller ...	1065	50	55
Carbon.....	39	Carbon ...	1105	85	105
"	40	Rumsey ...	1140	60	100
"	41	Three Hills ...	1075	75	55

FIGURE 3.



of the ash samples have been plotted against their softening temperatures. These points have been joined, so that the line represents the total range of temperature between the point of initial deformation and the fluid temperature. The length of the line below or above the oblique softening temperature line represents the softening and flowing intervals, respectively. The intervals, for convenience of comparison, have been plotted with respect to a horizontal zero line, and against their softening temperature at the bottom of the figure.

Another curve is appended, Figure 4, which shows the relation between the softening temperature of the ash of a coal and the amount of ash and the heating value of the same coal (Table V.). From this curve it will be noted that there is a general tendency for the softening temperature to rise with the amount of ash in the coal, but the ratio is not uniform. Attention is drawn to the fact that the coal samples with the lowest ash do not have the lowest softening temperature, nor do the coal samples with the highest ash have the highest temperature. There is even less relation between the softening temperature and the heating value, for though there is a small rise with the heating value, the sample with the highest value does not have the ash with the highest softening temperature, nor does the coal with the lowest heating value have the ash with the lowest softening temperature. The softening temperatures of the ash from these two are, in fact, close together, and the one with the highest heating value has a

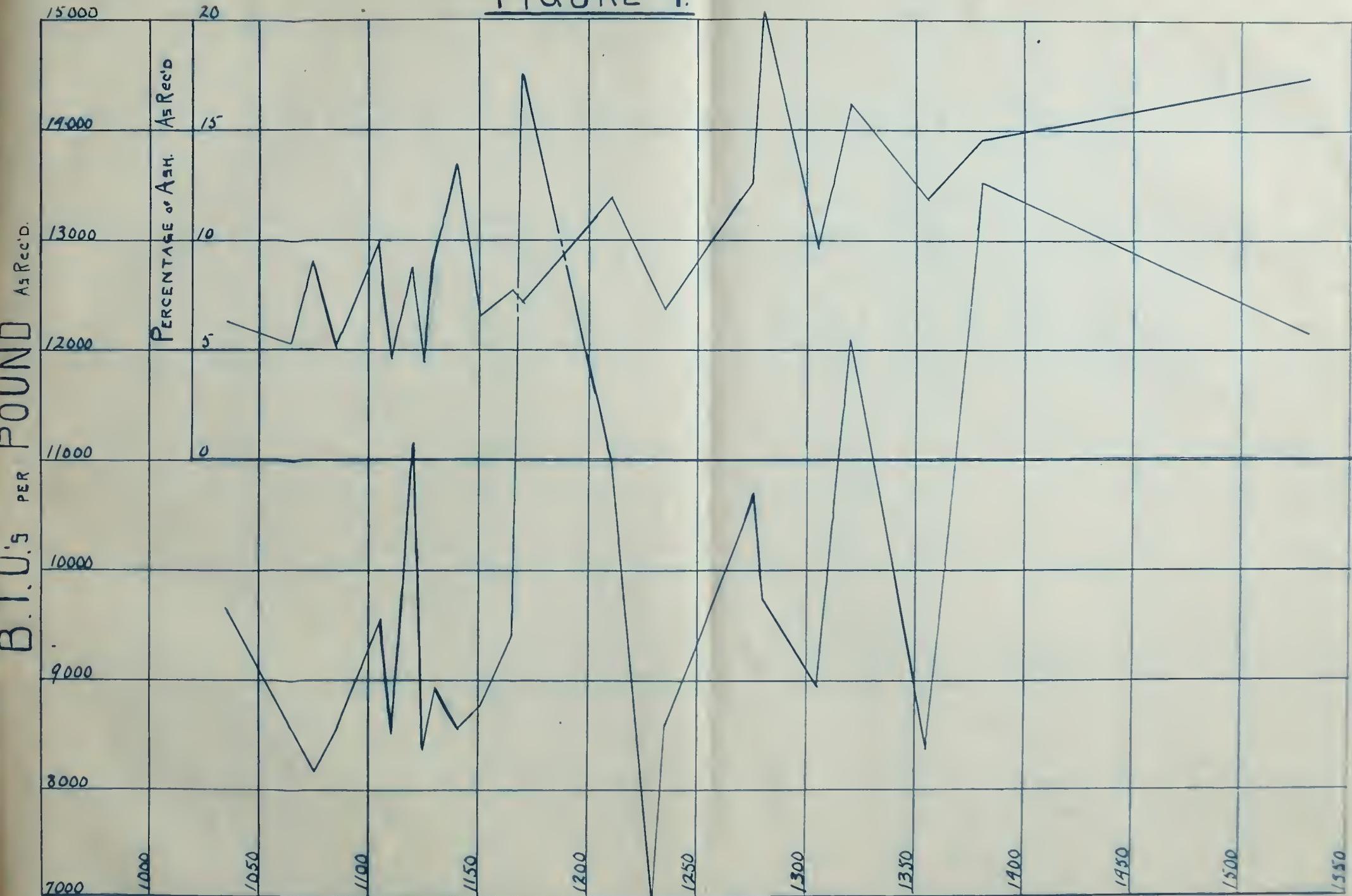
TABLE V.- SOFTENING TEMPERATURE OF ASH, ASH CONTENT AND HEATING VALUE OF ORIGINAL COAL*

Sample No.	Mine	Ash, %	Calorific Value, B.t.u. per lb.	Softening Temperature
37	Wayne ...	6.4	9,660	1035
38	Drumheller ...	5.3	9,845	1065
41	Three Hills ..	9.0	9,700	1075
25	Redcliff ...	8.5	8,135	1075
30	Tofield Coal	5.0	8,550	1085
32	Canadian Dinant	4.8	8,400	1085
39	Carbon ...	9.9	9,610	1105
33	Spicer ...	4.6	8,525	1110
14	Saunders ...	8.4	11,385	1110
11	Mercoal ...	8.6	11,115	1120
31	Dobell ...	4.5	8,350	1125
34	Ardley ...	8.9	8,940	1130
40	Rumsey ...	13.1	8,560	1140
21	Galt ...	9.3	10,930	1140
27	Penn ...	6.5	8,725	1150
35	Alix ...	7.7	9,460	1165
5	Canmore ...	7.2	14,480	1170
28	Twin City ...	6.1	8,840	1170
12	Foothills ...	11.9	10,920	1210
22	Coalhurst ...	9.8	10,980	1210
24	Pakowki ...	8.2	6,950	1230
29	Cardiff ...	6.7	8,525	1235
23	Magrath ...	12.5	10,740	1275
15	Lundbreck ...	16.8	11,190	1280
13	Sterling ...	20.3	9,730	1280
26	Pembina ...	9.6	8,995	1305
2	Luscar ...	16.1	12,510	1320
36	Big Valley ...	11.8	8,375	1355
4	Brazeau ...	14.5	13,505	1380
6	Hillcrest ...	17.4	12,200	1525
1	Blue Diamond	16.7	12,585	1550+
7	Greenhills ...	13.8	12,915	1550+
3	Cadomin ...	14.9	12,640	1550+

* The figures given under ash and calorific value above are the average of the analyses made in the laboratory of the Scientific & Industrial Research Council of Alberta on the coal samples as received.

All the values given above are not plotted on Figure 4; and, generally, where two samples have the same softening temperature, one is either omitted entirely, or given a slightly higher temperature value to preserve the continuity of the curve.

FIGURE 4.



SOFTENING TEMPERATURE
DEGREES CENTIGRADE

lower softening temperature than the one with the lowest heating value. From this evidence it can be stated that neither the ash content nor the heating value of the coal can be taken as an index of the softening temperature.

An inspection of Figure 3, in which the bituminous coals are plotted with dotted lines, shows that they have on the average a higher softening temperature than the lignites or sub-bituminous coals. With one exception (Canmore, 1170°C.) they are all above 1300°C.

The lignites range from 1035°C. to 1355°C., and the sub-bituminous from 1120°C. to 1280°C.

An analysis of the results shown in Table III. indicates, as would be expected, that, where two samples of coal were taken from one area, there is little variation between their softening temperatures. The range in typical cases is 25 to 60°C. Even allowing a maximum error of $\pm 10^{\circ}\text{C}.$, this only becomes 45° to 80° difference. These might be listed as follows:-

TABLE VI.- DIFFERENCE IN SOFTENING TEMPERATURES, SAMPLES FROM SAME AREAS

	Samples	Lowest Temp. $^{\circ}\text{C}$	Variation $^{\circ}\text{C}$	%	Seam
LIGNITES.....	Penn, Twin City.....	1150	20	2	Same
	Spicer, Dinant.....	1085	25	2	"
	Tofield, Dobell.....	1085	40	4	"
	Alix, Ardley.....	1130	35	3	"
	Galt, Coalhurst.....	1140	70	6	"
	Wayne, Drumheller.....	1035	30	3	#1, #5
SUB-BITUMINOUS	Carbon, Three Hills, Rumsey.	1075	65	6	Not known
	Mercoal, Foothills, Sterling	1120	160	14	Not known
BITUMINOUS....	Luscar, Cadomin.....	1320	180	14	Not known
	Greenhill, Hillcrest	Former	infusible		Same

In the case of the lignite coals the percentage variation obtained above compares favorably with the variation secured in determining the proximate analysis and the heating value of different coals from the same field, so that it might be justifiable to take the mean of those at hand as typical of the area till more data is secured.

The range of the softening temperatures in the bituminous and sub-bituminous areas is very great, but here the mountain-building forces have acted on the coal beds and caused different effects in places that are quite close together. This may account for the wide variation in the softening temperatures, or it may be due to the fact that the samples are from different seams. This is thought to be the case as far as the samples from the Coalspur area are concerned. Luscar and Cadomin mines, however, are but a few miles apart, and may be on the same seam, support being given to this theory by the fact that they have approximately the same ash content. The wide variation in the softening temperatures of their ash, on the other hand, suggests that they are on different seams, and this is supported by the next point discussed below.

Another point of interest is that the flowing interval would also appear to be typical of the seam. (See Table VII., next page.) Dinant and Spicer, with a difference of interval of 70°C., are the only exceptions to the rule among those that are known to be from the same seam, and these, it will be remembered, were also

different in color. The wide range between Cadomin and Luscar is a further argument that these two are on different seams.

TABLE VII.- DIFFERENCE IN FLOWING INTERVAL OF ASH SAMPLES FROM THE SAME SEAM

Samples	Difference	Exception	Difference
Penn, Twin City ...	0°C.	Cardiff	60°C.
Galt, Coalhurst ...	0
Tofield, Dobell ...	5
Alix, Ardley ...	0
Spicer, Dinant ...	70
*Carbon, Rumsey ...	5	Three Hills	50°C.
Luscar, Cadomin ...	100+

* These three are possibly from the same seam.

In connection with the sample from Cardiff, attention is drawn to the fact that it has a different color from the typical Edmonton samples, that there is a difference in the softening temperature of 130°C., or 11%, and that there is a difference in the flowing interval. All three facts point to the conclusion that Cardiff coal is not from the same seam that is being mined in Edmonton. There are also slight differences in the proximate analyses of the Cardiff and Edmonton coals. If further work confirms this indication, it would be a basis for suggesting that the Edmonton area be divided accordingly.

Another interesting point is that the seam mined at Pembina and Big Valley are both at the top of the same formation (Edmonton), and lie in the strata just below the Paskapoo series.

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Tofield, Dobell ...	5
Alix, Ardley ...	0
Spicer, Dinant ...	70
*Carbon, Rumsey ...	5	Three Hills	50°C.
Luscar, Cadomin ...	100+

* These three are possibly from the same seam.

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Luscar, Cadomin ...	100+

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Another interesting point is that the seam mined at Pembina and Big Valley are both at the top of the same formation (Edmonton), and lie in the strata just below the Paskapoo series.

From this it was concluded that they were in all probability the same seam. Confirmation is lent to this view by the fact that the softening temperatures are only 50°C. apart, which is less than 4% variation; and that the flowing intervals are 20° and 25°C., respectively; both of which are indications of the same seam.

These results may be a series of happy chances, but the apparent relation between color, softening temperature, and flowing interval of ash samples from the same seam is considered to be worthy of future study. If a relation could be established, it would be of great aid to the geologist in correlating seams and strata in Alberta. Some of the above information was, in fact, considered as collateral evidence by Dr. Allan in drawing up his recently issued map of Alberta coal areas.

C6nclusions Based On Constitution

Observation of a number of isolated facts suggests that the manner in which the ash-forming matter occurred might be of considerable importance. These included Rickett's second point in his report to the A.S.T.M., viz., the manner in which the ash-forming material exists in the coal; Sinatt's statement that the residue from ignited ankerites does not melt below 1425°C.; the fact that some bituminous coals, when ignited in small lumps, appeared to form several species of ash; the fact that similar species could be detected in examining the powdered

ash from bituminous coals under the microscope; and, in both cases, the presence of dark-colored matter that retained this color after prolonged heating in oxygen was noticed especially.

All these things pointed to the existence in the ash of distinct constituents, and it was decided to ignite the coal in small pieces without powdering, and to try and separate the components mechanically, if possible.

The first work was done with pieces of about 1/8-in. in size, and, later, with 1/4-in. pieces. The method adopted was to sift these free of dust and fines, and to heat them at a low temperature till oxidized, and later to heat them to about 800°C., as in the normal method of preparing ash. The pieces were heated on silica dishes, and as far as possible no piece of coal was allowed to touch any other piece. The dish was put in a cold furnace, and the slow heating prevented the formation of coke. On removal of the dishes from the furnace, several distinct varieties of ash were apparent, based on the colors which they displayed; and these could be separated by careful picking. In no case could a complete separation be obtained, for the particles were in all cases very fragile, and often two or three species would appear in a single lump. The pieces of ash had as a rule the size and shape of the lump of coal that had been ignited, and though some were quite hard, others fell to pieces on being touched.

The purest pieces of each general species were collected and examined, physically and chemically. Coal containing these

was divided into four portions by heavy solutions in an attempt to isolate the source of the species; and where enough was obtained, the softening temperature was determined.

Four general species of ash constituents were recognized:-

Group A - pure white, grey, or, occasionally, hard and pink in color;

Group B - brown, or earthy in color;

Group C - red in color (the shade of dried blood);

Group D - light red, pink, or salmon shade in color.

The results of the different tests are presented in tabular form in Tables VIII. and IX. The ash is first divided into species and sub-species on the basis of its color and state. The relative amounts of each species from coal with different gravities is indicated, and it is to be noted that it was the coal that was separated by the heavy solutions. The separation of all the mineral matter with high gravity into that part of the coal with a gravity over 1.55 is not complete, because small pieces sometimes occur which are formed of a large amount of coal with a little extraneous matter attached. Such a piece acts with a gravity in between the two, and in this way small amounts of mineral matter appear in the ash from coal showing gravities much less than the extraneous matter itself. This separation can not be made complete without very fine division of the coal, but then the heavy solution does not easily wet the particles, and the method will not work satisfactorily for this reason.

The chemical examination tabulated was purely qualitative,

and was carried out in order to identify the sources of the mineral matter whose presence was indicated by the separations made with the heavy solutions. It was generally satisfactory, but failed to distinguish between shale and sandstone.

The amounts of ash available were in some cases very small, and usually were contaminated with other constituents, so that the fusion tests are not by any means complete; but enough was done to give a general idea of the softening temperatures of the different species.

Pieces of extraneous matter were picked out of Sterling ash by hand, but could not be identified by any physical property, though differences became apparent at once, following ignition.

A study of the charts indicates the presence of low-fusible compounds in the ash from bituminous coals, one class having a softening temperature of $1405^{\circ}\text{C}.$, and another, $1140^{\circ}\text{C}.$, while the majority of the ash is infusible at $1500^{\circ}\text{C}.$ It is submitted that the low-fusing constituents are those which form a certain amount of clinker in the burning of coals with an infusible ash. It does not follow that these compounds will melt at the temperatures given, for the action of the other elements may lower their softening temperature to some extent, but the figure given will likely be the maximum temperature required to soften these particular constituents. It is interesting to note in this connection that the figure, $1405^{\circ}\text{C}.$, given above, applies to

ankerite, for whose softening temperature Sinatt gives 1425°C.

If further investigation does not disprove this theory, it follows that the amount of clinker formed will depend to a large extent on the amount of constituents present that have a low softening temperature, since they are not intimately mixed in furnace practice with the high-fusing components. Where this does occur, as in the burning of powdered coal, the use of an infusible ash as determined by the standard method, should give practically no trouble at all.

There is also here an explanation of the fact that coal can be washed without materially affecting the softening temperature of its ash, for it is possible that the right proportion of shale, ankerite, pyrite, etc., might be removed, so that the ash that is left would have a softening temperature that was the same as, above, or below that of the original ash. (See Chapter VII., "Effect of Washing Coal on the Softening Temperature of Its Ash.")

Titanium is found only in the extraneous ash from shale, and bone, and we should therefore expect it to be high in ash with a high proportion of these substances. It is, in fact, high in high ash bituminous coals, showing that a large proportion of their ash comes from these sources.

It is apparent, too, that calcium, iron, magnesium, and probably silica, are present in the ash as free oxides, and the behavior of these when being heated may be different from the

same oxides in combination as salts. It is pointed out that the iron in the ankerites has a distinctly different color from that in intrinsic ash, and this explains the presence of large amounts of iron in the bituminous coal ash, which are generally of a grey color, while low ash coals with less iron are highly colored. This is the reason that color does not indicate the softening temperature in any reliable way, and presumably the small, dark-colored grains recognized under the glass in the bituminous coals are particles of brown ankerite residue.

TABLE VIII.- CONSTITUENTS OF BITUMINOUS COAL ASH, CADOMIN & LUSCAR

Species..	A1	A2	B	C	D
Color.....	White	Gray	Brown	Red	Pink
State.....	Powder	Hard	Powder	Flakes	Powder or Skeletons

Relative Amount of Each Ash Species in Coal of Specific Gravity Noted*

*S.G. of Coal					
Over 1.55	Large	Large	Large	Small	Nil
1.45-1.55	"	"	"	Very small	Trace
1.35-1.45	"	"	"	Trace	Small
Under 1.35	"	"	Nil	"	Very large
Probable source...	Mineral matter and coal		Mineral matter	Mineral matter	True coal

Qualitative Chemical Examination

Elements Present					
Si.....	Heavy	Heavy	Nil	Nil	
Al.....	"	"	"	"	Contains all elements except
Ti.....	"	"	"	"	
Fe.....	Trace	Trace	Heavy	Very heavy	
Ca.....	Nil	Nil	Very heavy	Trace - heavy	Titanium
Mg.....	"	"	Heavy	Light	
S.....	Trace	Trace	Trace	Trace	
P.....	"	"	"	"	
Source....	Coal, bone, shale & sandstone	Ankerite, 2Ca.Fe.Mg.	Variety of Ankerite, Pyrite, Limonite		True coal
S.G. of Source..		3.0	Ank. = 3.0 Pyr. = 5.0 Lim. = 3.8		Less than 1.35

Fusion Tests

Ash Species and Coal	Softening Temperature	Softening Interval	Flowing Interval	
A1 & A2 Cadomin or Luscar	1500+°C.	400+°C.	
B Cadomin	1405	360	50°C.) Very
B, C, & D Luscar	1370	355	65) viscous
C & D Cadomin	1140	105	20	
Cadomin Ash	1550+	400+	
Luscar Ash.....	1320	210	100	

* Coal as separated by heavy solutions

TABLE IX.- CONSTITUENTS OF STERLING COAL ASH (SUB-BITUMINOUS)

Species...	A				B	C	D
	A1	A2	A3	A4			
Color.....	White or	grey	Pink	Brown	Red	Pink	
State.....	Powder or hard		Hard	Powder	Flakes	Powder	

Qualitative Chemical Examination

Elements Present	Si	Al	Ti	Fe	Ca	Mg	S	P		
	None	None	None	None	Very heavy	Trace	Trace	Trace	None	All elements present except Titanium
						Trace	Trace	Trace	Trace	
							Trace	Trace	Trace	
									(Very Heavy)	
Source.....	Calcite				Bone, shale, sandstone and coal				Ankerite	True coal

Fusion Tests

Source of Ash	Softening Temperature, °C.	Softening Interval, °C.	Flowing Interval, °C.
A ₁	Infusible at 1500		
A ₂ , A ₃ , A ₄	Infusible at 1500		
B.....	1405	360	50
Sterling Ash, No.1	1390	240	75
Sterling Ash, No.2	1280	170	65

CHAPTER VI.- RELATION BETWEEN RESULTS
OBTAINED IN THE LABORATORY AND
CLINKER FORMATION IN PRACTICE

Relation Of Softening Temperatures
To Clinker Formation

Sinatt, working in England, determined the softening temperature of the ash from different horizons in the same seam, and found in some cases wide differences in the softening temperature. This is in line with the quotation from Bone given at the head of the previous chapter. In one particular case quoted, the variation was from 1195 to 1440°C.. The high figures generally applied to horizons with a high ash content, and there is therefore the suggestion that these portions carried a considerable amount of siliceous matter. In other seams, however, the horizons showed a difference of only 35 or 40°C..

The same worker separated some bituminous coal into its four ingredients, as defined by Dr. Stopes, and determined the softening temperature of the ash from these, finding again a wide variation, usually from 200 to 250°C.. In each case the fusain was the lowest, and the durain the highest.

These results were all obtained with the furnace described above, and the comments made in that connection apply to these results.

Besides the variations in the horizons of a coal seam and in the different portions of the coal itself, there are the

the pieces of extraneous ash-forming matter; though it should be noted that the larger of these portions of shale, bone, etc., pass through the fire as inert matter, and remain for the most part unfused.

It follows from these considerations that the softening temperature, as determined by the furnace, is only truly representative of low ash coals where the ash content is finely divided and more or less uniformly distributed through the coal mass, this being the condition most nearly approximating the standard method, where the whole of the ash is finely powdered and intimately mixed before fusion. This procedure favors the constituents present in the greatest proportion, whether they are high or low fusing in their nature; so that, with coals containing a high proportion of refractory matter in the ash, the results shown will have to be modified. This is common among the bituminous coals of this province, which have a high ash containing a high percentage of siliceous matter; and the method does in fact fall down with these; for, though they appear infusible by the standard method, they do form a small amount of clinker, both hard and soft. This is explained by the fusion of low-softening bodies, the presence of which is demonstrated above by Sinatt's work, and ~~which he mentioned~~ again above under the heading "Constitution of the Ash." Though the method does not give an exact interpretation of this class of coals, it is satisfactory as indicating that they will give little or no trouble with clinker.

It follows, therefore, that the percentage of ash does not greatly enter into the interpretation of the results obtained by the standard method in some cases; for, since the low ash coals are most accurately represented by the method, it is reasonable to suppose that almost the whole of the ash would soften at the indicated temperature, while in high ash coals, since the ash-forming matter is not well mixed, some of the higher fusing portions will not be likely to soften at the temperature indicated. The mass effect of a large amount of ash, in the heat that it absorbs, will lower the temperature also, with the consequent formation of less clinker. It would seem, therefore, that the amount of clinker formed by low and high ash coals with the same softening temperature and softening interval will differ only if the high ash coal is formed of constituents with a fairly constant softening temperature; but that if the high ash coal is a mixture of high and low fusing constituents, which will usually be the case, the amount of clinker formed by the two coals will not differ greatly.

The intervals are important in interpreting the results obtained by the standard method, as well as the softening temperature; for they are an indication of the relative viscosity of the clinker formed. Obviously the ash which flows the least, even if soft, will not form such troublesome clinker as the one which is more fluid, which will flow around the infusible portion and either dissolve it or engulf it bodily, to

form large, hard clinkers. In two coals, therefore, which have little or no variation in their softening temperature, the one which has the longest flowing interval will give the least trouble.

Reference has been made at various stages to the temperature of the fuel bed, since this is obviously an important factor in the formation of clinker, and this will now be considered.

Relation Of Calorific Intensity To Clinker Formation

In the preceding portions of this paper it has been assumed that there is a relation between the heat developed in the fuel bed and the amount of clinker formed. This was based on the observation that at higher ratings, when the temperature of the fuel bed was higher, there was more clinker from the same coal, and on the fact that of two coals, which seemed superficially to be very much alike, the one with the greatest heating value generally formed the most clinker. Closer study of this phase of the question has upheld this theory. (See Bailey, E.G.: Power; above, Chapter II., "Historical"; and Ricketts' report to the A.S.T.M., noted above in Chapter I..)

As a result of these considerations it was decided to try to obtain as far as possible the value of the calorific intensity of the coals sampled. In this way a comparison could be made between the temperature required to soften the ash, as represented by the softening temperature, and the maximum heat to which it would be subjected, as represented by the calorific intensity.

The calculation of the calorific intensity is based on the ultimate analysis, and only a limited number of these were available, but as many as possible were estimated. Each calculation referred to a particular coal, but the one whose analysis most nearly represented the average of its field was selected as the basis for the calculation. The average analyses were based on the results obtained in the analytical laboratory of the Scientific and Industrial Research Council of Alberta. The ultimate analyses used were taken from Bulletin 25 of the Department of Mines, Ottawa, and the Third Annual Report of the Alberta Research Council.

The calorific intensity of nineteen coals were calculated. The method, as applied to a particular coal, consisted in estimating the composition of the flue gases that would be formed by burning a coal whose composition was determined by its ultimate analysis; and, by means of the known specific heat of the gases formed, the amount of heat required to raise a gram molecule of the theoretical flue gas to different temperatures was obtained. The heating value of the coal in gross calories was corrected for the amount of water vapor formed and condensed in the bomb determination, and for the amount of hygroscopic water which must be vaporized in a furnace, giving a value for the total heat available to raise the temperature of the combustion gases, in net calories.

To obtain the heat available per gram molecule of flue gas, the net calorific value was divided by the number of gram

molecules of flue gas formed. Comparison of this value with the values showing the amount of heat required to raise a gram molecule of the flue gas to different temperatures gave the maximum temperature attainable for that particular coal.

Since the coal is never burned in practice with the theoretical amount of air, the calculation was carried out on the supposition that 50% excess air would most nearly represent the actual conditions.

The amount of oxygen in the coal affects the amount of oxygen required from the air, and so the total volume of the flue gases for the excess of nitrogen and oxygen is equal to 6.5 times the volume of the oxygen required from the air. For this reason, the calculated temperature will be in error by an amount depending principally on the error in the determination of the oxygen. The percentage of oxygen given by the ultimate analysis may be in error by the sum of the errors of the determination of the other elements in the coal, since it is obtained by difference, and the error may amount to 5% of the value so found.

In the tables given in this connection, the heating value is reported in the usual way: viz., B.t.u's (calories per gram, multiplied by 1.8).

An example of the method of calculation, as applied to Penn coal, is given below.

Example Of The Method

(Data from Bulletin 25, Mines Branch, Ottawa, p. 49, Sample M-45.)

Carbon.... 50.1%	Moisture..... 23.7%
Hydrogen.. 6.0	contains H ₂ 2.6%
Oxygen.... 36.4	Net Hydrogen in coal 3.4%
Nitrogen.. 1.0	Calories per gram, gross, = 4820 ± 10
Sulphur... 0.3	Correction for oxygen = 5 (Note 1)
	Correction for water = <u>180</u> (Note 2)
	185... 185
	Calories per gram, net... = 4635

In 1000 grams of coal there are:

$$501/12 = 41.7 \text{ gram atoms C, forming } 41.7 \text{ gm. mol. CO}_2$$

$$60/ 2 = 30.0 \text{ gm. mol. H}_2\text{, forming } 30.0 \text{ gm. mol. H}_2\text{O}$$

Oxygen Required:

For 41.7 gm. mol. CO ₂	41.7 gm.mol. O ₂
For 30.0 gm. mol. H ₂ O.....	<u>15.0</u> " " "
Total required.....	56.7 " " "
Oxygen in coal = 364/32	<u>11.4</u> " " "
Oxygen required from air.....	<u>45.3</u> " " "
Nitrogen from air = O ₂ X 4 = 181.2 gm.mol. N ₂	
Nitrogen in coal = 100/28 = .4 " " "	
For 50% excess air add.....	<u>90.6</u> " " " & <u>22.7</u> gm.mol.O ₂
	272.2 " " " 22.7 " " "

Composition of The Flue Gases:

Carbon dioxide.....	41.7 gm.mol. or 11.4%
Water vapor.....	30.0 " " 8.2
Oxygen.....	22.7 " " 6.2
Nitrogen.....	<u>272.2</u> " " <u>74.2</u>
	366.6 " " " 100.0%

The Increase in Heat Content of Gases (see below) is:- (Note 3)

G a s	1400°C.	1600°C.	1800°C.
Carbon dioxide..	16.22 K	18.93 K	21.62 K
Water Vapor.....	12.94 K	15.32 K	17.85 K
Nitrogen, Oxygen	10.16 K	11.80 K	13.48 K

K = 1000 cal.

The Increase in Heat Content of Theoretical Flue Gas is:-

G A S	%	1400°C.	1600°C.	1800°C.
CO ₂	11.4	1.85 K	2.16 K	2.46 K
H ₂ O....	8.2	1.06 K	1.26 K	1.46 K
N ₂ & O ₂ ..	80.4	8.17 K	9.48 K	10.82 K
Total...	100.0	11.08 K	12.90 K	14.74 K

On burning 1000 grams of coal there are available... 4635 K
Correction for water content = 237×0.585 K..... = 140 K (Note 4)
Net heat available is..... 4495 K
" " " per molecule of flue gas is $4495/366.6 = 12.25$ K

From table, "Increase in Heat Content of Theoretical Flue Gas",
 12.90 K - 11.08 K = 1.82 K, and 12.25 K - 11.08 K = 1.17 K.

Assuming this function to be linear, we obtain from the ratio:

$$\frac{1.17 \times 200}{1.82} = 129, 1400 + 129 = 1529 \text{ as the maximum temperature.}$$

On analysis, this theoretical flue gas loses its moisture content,
and gives the following values:-

Carbon dioxide.....	12.3%
Oxygen.....	6.7%
Nitrogen.....	<u>81.0%</u>
	100.0%

Notes On The Method

1. Correction For Oxygen.- In the determination of the calorific value of the coal there is a loss of volume due to the union of oxygen with the hydrogen in the coal to form water. This takes place at constant volume, so that the energy gained, represented by RT , expressed in calories, is $1.95 \times (273 + 20) = 580$ calories per molecule of oxygen, or per 2 molecules of hydrogen. For the coals under consideration, this represents 4.2 to 6.0 calories per gram, and a uniform correction of 5 calories has been made.

2. Correction For Condensed Water Vapor.- In the determination of the calorific value, the water vapor formed is condensed, giving up its heat of vaporization. At 20°C . this equals 585 calories per gram of water formed. This amount is obtained from the net hydrogen in the coal, each gram of which forms 9 grams of water. In the example above:-

1 gram of coal contains 0.034 gms. H₂, forming 0.306 gms. water.

Total heat liberated on condensation = 0.306 X 585 = 180 calories.

This correction varies from 170 calories in the high-moisture coals (305 B.t.u.), to 225 calories (405 B.t.u.) in the bituminous coals.

3. Increase In Heat Content Of Gases.- The values for the increase in heat content are obtained from the following data:-

- (1) Lewis & Randall: Thermodynamics, 1923, p. 80, and
(2) Goodenough, S.A., & Felbeck, G.T.: An investigation of the maximum temperatures and pressures attainable in the combustion of gaseous and liquid fuels, University of Illinois, Bulletin 139, March, 1924, p. 106.)

Specific Heats of Gases

$$\text{O}_2 \text{ and N}_2 \dots \begin{aligned} (1) \quad C_p &= 6.50 + 1.0 \times 10^{-3} T \\ (2) \quad C_p &= 6.93 + 0.389 \times 10^{-6} T^2 \end{aligned}$$

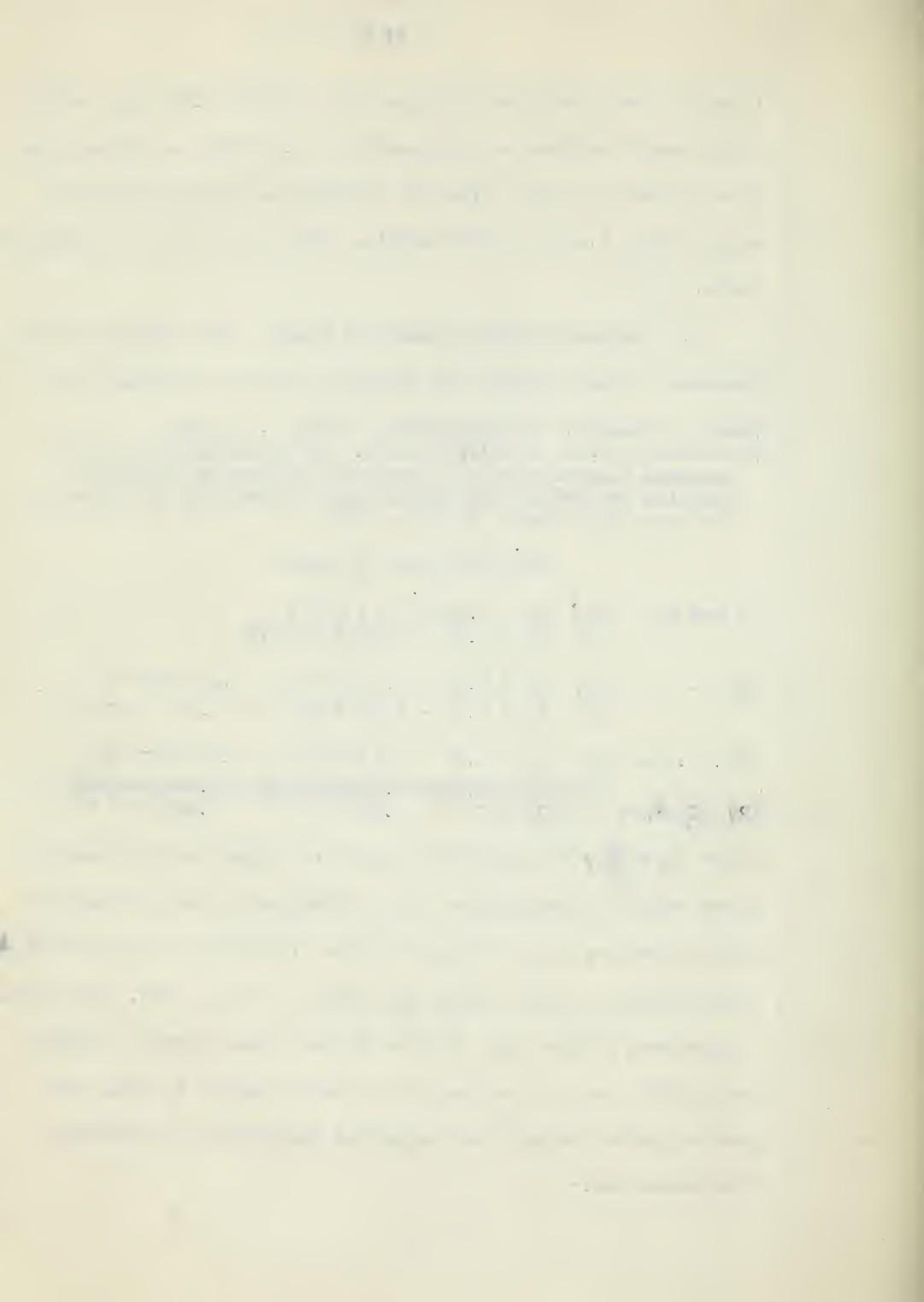
$$\text{H}_2\text{O} \dots \begin{aligned} (1) \quad C_p &= 8.81 - 1.9 \times 10^{-3} T + 2.22 \times 10^{-6} T^2 \\ (2) \quad C_p &= 8.33 - 0.497 \times 10^{-3} T + 1.37 \times 10^{-6} T^2 \end{aligned}$$

$$\text{CO}_2 \dots \begin{aligned} (1) \quad C_p &= 7.00 + 7.1 \times 10^{-3} T - 1.86 \times 10^{-6} T^2 \\ (2) \quad C_p &= 6.46 + 9.12 \times 10^{-3} T - 4.05 \times 10^{-6} T^2 + 0.63 \times 10^{-9} T^3 \end{aligned}$$

Since $C_p = \frac{dQ}{dT}$, the quantity of heat to raise the gas through a given range of temperature, can be obtained by integrating the above formulae; e.g., for O₂ (2), $Q = 6.93(T_2 - T_1) + 0.13 \times 10^{-6}(T_2^3 - T_1^3)$.

Substituting in this formula the limits for T₁ = 20°C. (the normal temperature of the air), and for T₂ the values 1200°C., 1400°C., and 1600°C., etc., the quantity of heat required to raise one gram molecule through the respective temperatures is obtained.

The values are:-



Gas	1200°C.	1400°C.	1600°C.	1800°C.
CO ₂	13.57	16.22	18.93	21.62
H ₂ O....	10.77	12.94	15.32	17.85
N ₂ , O ₂ ..	8.58	10.16	11.80	13.48

The values given by both (1) and (2) are the same within the limits of experimental error. Goodenough's formulae are slightly lower, and have been used in these calculations.

4. Correction For Water Content.- The water content of the coal is vaporized in the furnace, and this requires 585 calories per gram (20°C.) which can not be used to raise the temperature of the flue gases. In the determination of the heating value this takes place, but the vapor is condensed, giving its heat up again.

This correction, applied to commercial coals is:-

Percentage of water X 10.56 = B.t.u. per pound. (Latent heat of vaporization of uncondensed steam at 62°F. = 1056.7 B.t.u., according to Marks & Davis.)

5. Sources of Error Not Corrected For.- The volume of gas produced by burning the sulphur in the coal is neglected, and also the amount of oxygen necessary to combine with it. The heat given out by the oxidation of the sulphur and nitrogen to acids is corrected for in the determination of the heating value. The volume of sulphur in Alberta coals is in every case quite small -- generally about 0.3%.

The composition of the air is taken as 20% Oxygen and 80% Nitrogen, which is not the exact case, but both have the same

specific heat.

The amount of water vapor present in the air is not estimated.

In practice there is always an amount of carbon monoxide formed, varying with the air supply. This tends to lower the amount of oxygen required; and lower the amount of heat available, owing to incomplete combustion. This was not considered in the calculation.

The amount of heat lost through radiation by the furnace walls, by fusion of ash, and the amount necessary to heat the ash up to the temperature reached were all neglected. These will vary with the temperature and the ash content of the coal.

There is probably also some dissociation of the gases at the temperatures reached, which may alter the amount of heat available.

It rarely happens in actual practice that the excess of air is maintained at 50% on the average, so that for all these reasons the maximum temperature will never be approached in furnaces as operated. The value obtained is for an ideal furnace, to which commercial ones only approximate. Actual measurements of a brisk fire of Penn slack gave a value of 1300°C. (optical pyrometer), compared to the value of 1530°C. obtained by calculation.

It is thought that the figures obtained for the calorific intensity give values which are quite comparable, since they apply

to an ideal furnace under identical conditions, and that they can be used for the purpose of comparing the coals.

Estimation Of The Calorific Intensity Where No Ultimate Analysis Was Available

A relation was sought between the calorific intensity so calculated, and either the proximate analysis or the heating value or a combination of the two for the coals considered, in order to get a comparative figure for the coals, the ultimate analyses of which were not available.

After many comparisons it was found that the ratio between the calculated intensity and the net B.t.u. as received made the nearest approach to a constant. On plotting these values (see Figure 5.) and drawing a theoretical curve, it was found that none of the nineteen varied by more than 2%. This variation is considered to be due to experimental error, and compares favorably with the percentage error in the calculation of the calorific intensity, and the allowable error in the estimation of the softening temperature (a tolerance of 30°C. for the same operator), so that the use of the curve to obtain values for the calorific intensity of other coals seems justifiable.

To use it, the net calories must first be determined, and where no ultimate analysis was available, the net hydrogen content of the coal was taken from the analysis of a similar coal in the same area, to determine the correction for condensed moisture in the bomb. The correction for hygroscopic water was calculated

from the formula given above.

The water content of the coal was plotted against the total correction in the B.t.u. (See Figure 6.), and a theoretical curve drawn, and it was found that a fairly close relation existed between the two. This can be stated as follows:-

<u>Moisture, %</u>	<u>Correction in B.t.u. value, as received</u>
Above 12.....	$390 + 8 \times \text{percentage of moisture}$
Below 12.....	$390 + 12.5 \times \text{percentage of moisture}$

A table was prepared (Table X-a) showing the actual correction based on the net hydrogen in the coal and the water content, and the correction calculated by the formulae above, together with the difference in the two values. It is thought that these differences are small enough to warrant its use for estimating the net calorific value of a coal of which no ultimate analysis is available.

From the net calorific value, the calorific intensity can be estimated by the use of the curve in Figure 5. This was done, and a table was prepared (Table XI.) showing the estimated calorific intensity of all the coals from which ash samples had been taken. By the use of these curves the calorific intensity developed by a coal under the conditions outlined above can be estimated from its heating value and moisture content.

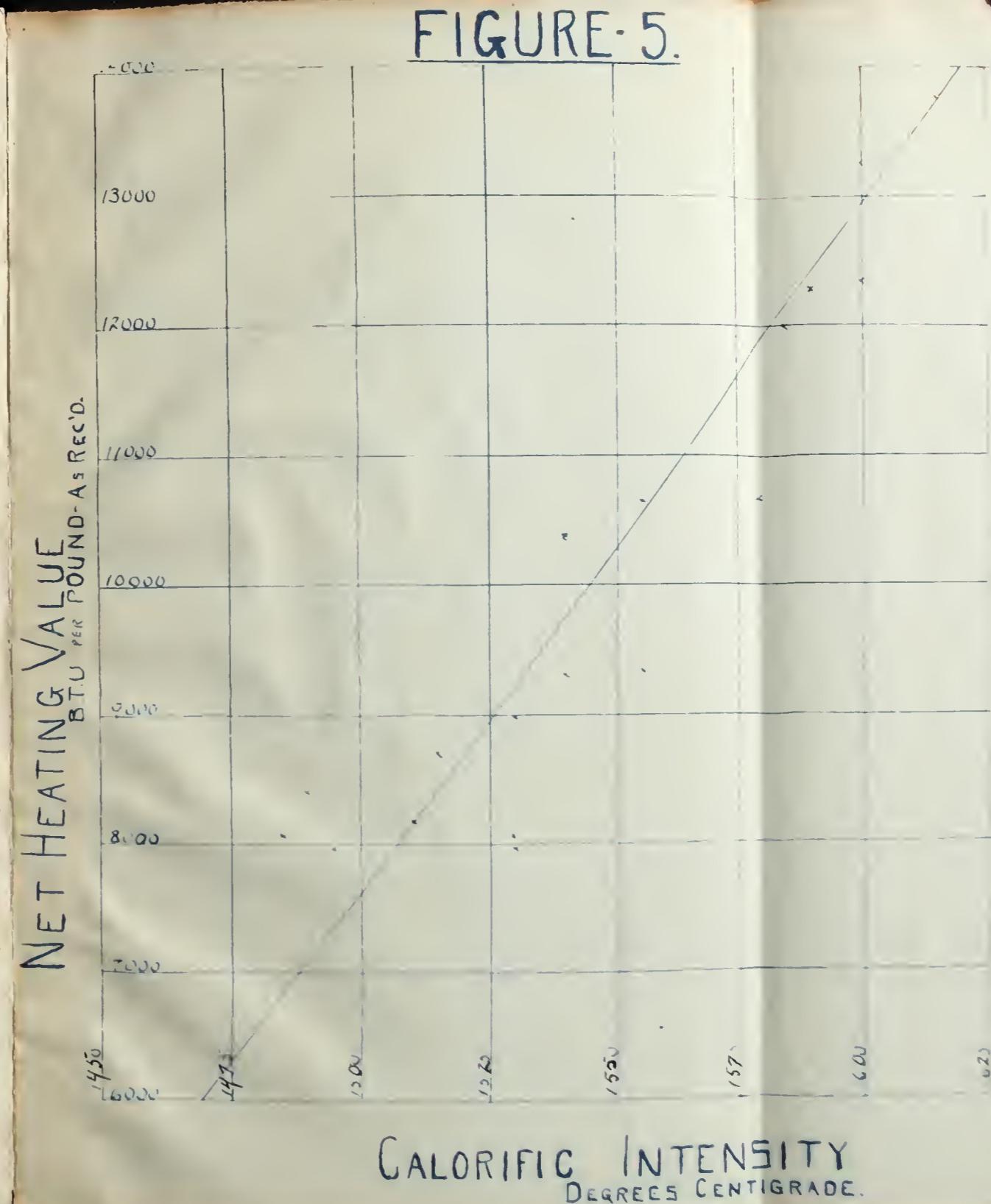
TABLE X.- DATA USED IN CALCULATING THE CALORIFIC INTENSITY*

M i n e	C %	H %	O %	N %	S %	Ash %	H ₂ O %	B.t.u. per lb., gross	B.t.u. per lb., net	Calorific Intensity, °C.
Twin City.....	52.1	5.9	34.7	1.0	0.3	6.0	23.8	8670	8075	1485
Pembina.....	53.9	5.5	29.3	0.8	0.2	9.7	18.2	8930	8400	1490
Cardiff.....	51.5	6.1	33.6	1.0	0.2	7.6	21.2	8570	7975	1495
Dobell.....	52.3	5.7	34.4	1.0	0.4	6.2	22.2	8740	8190	1510
Big Valley....	55.4	5.3	30.4	0.9	0.2	7.8	18.2	9210	8700	1515
Tofield Coal..	49.6	6.1	37.8	1.0	0.5	5.0	26.3	8580	7985	1530
Ardley.....	55.9	5.6	28.7	1.0	0.4	8.4	17.1	9530	8990	1530
Penn.....	50.1	6.0	36.4	1.0	0.3	6.2	23.7	8670	8085	1530
Wayne.....	58.5	5.3	27.8	1.3	0.4	6.8	13.9	9800	9305	1540
Coalhurst....	63.3	5.4	19.4	1.6	0.6	9.7	8.9	10890	10360	1540
Galt.....	62.9	5.5	20.0	1.5	0.5	9.6	9.8	10880	10370	1540
Drumheller....	57.3	5.7	29.6	1.2	0.4	5.8	18.6	9890	9330	1555
Saunders.....	65.7	5.2	21.4	1.0	0.3	6.4	10.8	11150	10645	1555
Mercoal.....	64.3	4.9	17.6	0.9	0.1	12.2	6.0	11110	10640	1580
Greenhill.....	72.0	4.4	7.0	1.0	0.5	15.1	0.9	12410	11995	1585
Blue Diamond..	74.2	4.0	3.7	1.3	0.3	16.5	0.9	12680	12295	1590
Brazeau.....	79.7	4.1	3.1	1.1	0.4	11.6	0.6	13690	13275	1600
Cadomin.....	73.0	4.4	6.1	0.9	0.4	15.1	0.7	12760	12335	1600
Canmore.....	82.8	4.2	4.5	1.6	0.7	6.2	1.9	14280	13885	1615

In three cases, figures are taken from nearby mines:-

Mountain Park was substituted for Cadomin;
Yellowhead Pass Coal & Coke, for Mercoal;
Lousana, for Big Valley.

* Data from Bulletin 25, Mines Branch, Ottawa; and Third Annual Report, Scientific & Industrial Research Council of Alberta.



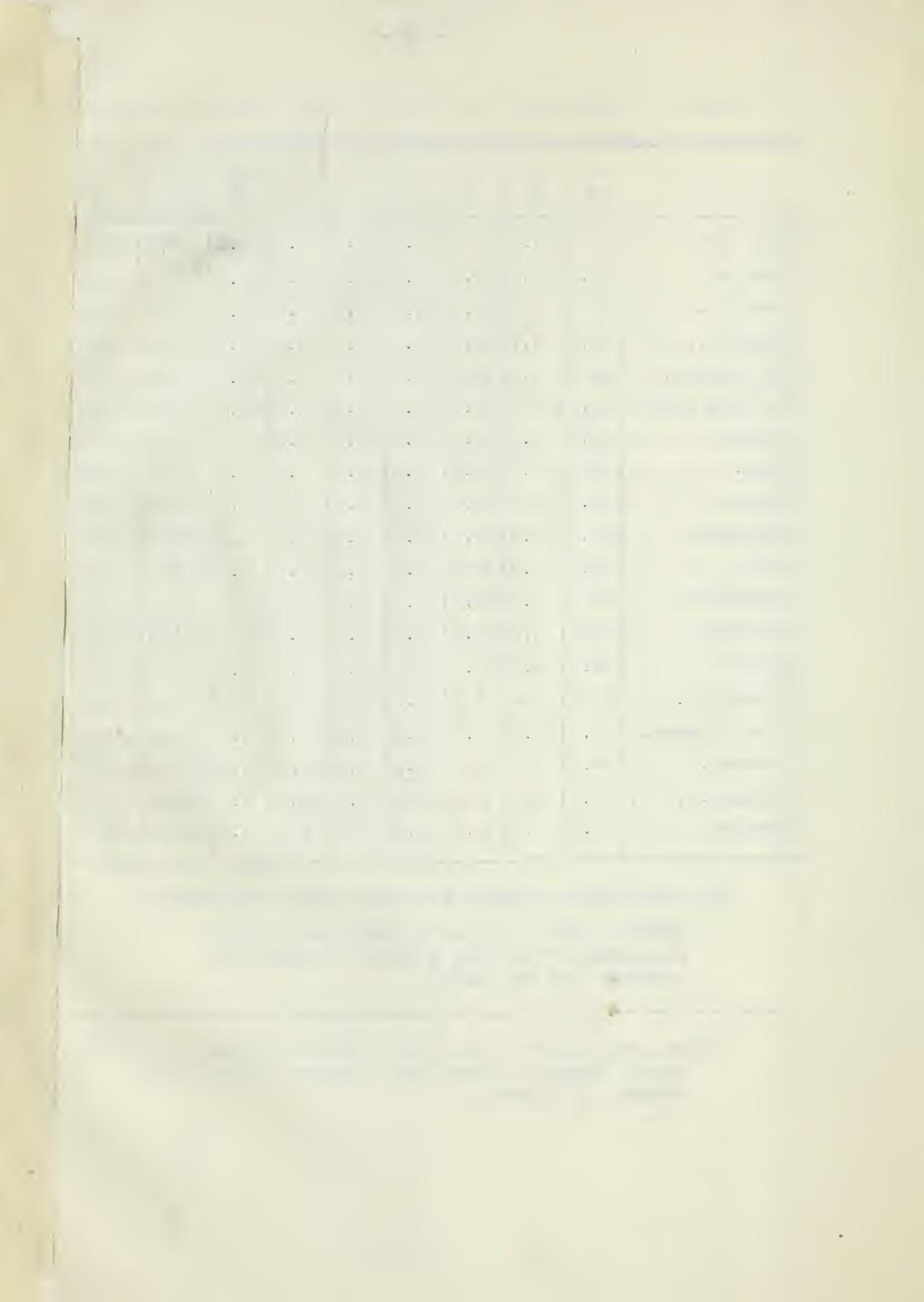


TABLE X-a.- ASH SAMPLES IN ORDER OF SOFTENING TEMPERATURE

Showing moisture content; B.t.u. correction (1) calculated on the percentage of hydrogen, and (2) by formula; and the difference in values of the B.t.u. corrections obtained.

Sample No.	Mine	Water, %	B.t.u. Correction		Difference (2) - (1)
			(1)	(2)	
37	Wayne.....	18.1	540	535	- 5
36	Drumheller....	19.0	560	540	- 20
41	Three Hills...	16.8	520	525	+ 5
25	Redcliff.....	26.1	585	600	+ 15
30	Tofield.....	27.0	600	605	+ 5
32	Can.Dinant....	27.0	600	605	+ 5
39	Carben.....	16.9	525	525	0
33	Spicer.....	26.6	595	600	+ 5
14	Saunders.....	8.5	480	495	+ 15
11	Mercal.....	7.8	485	485	0
31	Dobell.....	28.8	620	620	0
34	Ardley.....	20.7	580	555	- 25
40	Rumsey.....	19.7	550	545	- 5
21	Galt.....	10.4	535	520	- 15
27	Penn.....	24.5	590	585	- 5
35	Alix.....	18.4	560	540	- 20
5	Canmore.....	1.9	400	415	+ 15
28	Twin City.....	25.7	610	595	- 15
12	Foothills.....	7.4	485	480	- 5
22	Coalhurst.....	9.9	535	510	- 25
24	Pakowki.....	33.0	650	655	+ 5
29	Cardiff.....	25.5	625	595	- 30
23	Magrath.....	9.8	525	510	- 15
15	Lundtreck.....	6.7	470	475	+ 5
13	Sterling.....	7.3	470	480	+ 10
26	Pembina.....	19.9	535	550	+ 15
2	Luscar.....	3.7	455	435	- 20
36	Big Valley....	20.9	545	555	+ 10
4	Brazeau.....	1.8	425	410	- 15
6	Hillcrest.....	1.3	420	405	- 15
1	Blue Diamond..	1.3	405	405	0
7	Greenhill.....	2.1	425	415	- 10
3	Cadomir.....	2.4	440	420	- 20

FIGURE - 6.

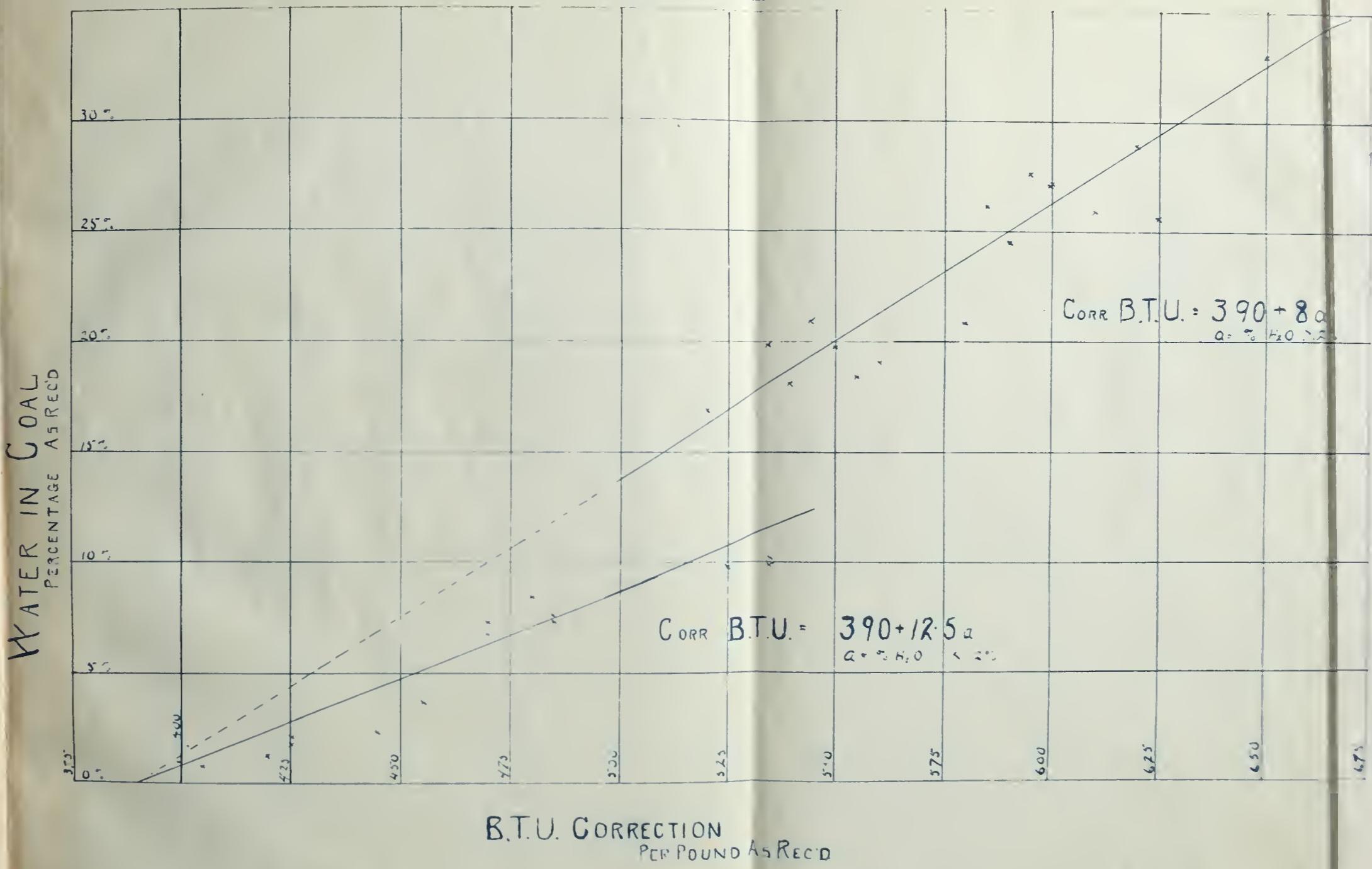


TABLE XI.- ASH SAMPLES IN ORDER OF SOFTENING TEMPERATURE,
SHOWING ESTIMATED CALORIFIC INTENSITY

Sample No.	Mine	Water, %	B.t.u. per lb.,		Calorific Intensity	Softening Temperature	
			Gross	Net		°C.	Order
37	Wayne.....	18.1	9,660	9,120	1530	1035	1
38	Drumheller...	19.0	9,845	9,285	1530	1065	2
41	Three Hills..	16.8	9,700	9,180	1530	1075	3
25	Redcliff.....	26.1	8,135	7,550	1500	1075	4
30	Tofield.....	27.0	8,550	7,950	1505	1085	5
32	Can. Dinant..	27.0	8,400	7,800	1500	1085	6
39	Carbon.....	16.9	9,610	9,085	1525	1105	7
33	Spicer.....	26.6	8,525	7,930	1505	1110	8
14	Saunders.....	8.5	11,385	10,915	1560	1110	9
11	Mercoal.....	7.8	11,115	10,630	1555	1120	10
31	Dobell.....	28.8	8,350	7,730	1500	1125	11
34	Ardley.....	20.7	8,940	8,360	1515	1130	12
40	Rumsey.....	19.7	8,560	8,010	1505	1140	13
21	Galt.....	10.4	10,930	10,395	1550	1140	14
27	Penn.....	24.5	8,725	8,135	1510	1150	15
35	Alix.....	18.4	9,460	8,900	1525	1165	16
5	Canmore.....	1.9	14,480	14,080	1620	1170	17
28	Twin City.....	25.7	8,840	8,215	1510	1170	18
12	Foothills....	7.4	10,920	10,435	1550	1210	19
22	Coalhurst....	9.9	10,980	10,445	1550	1210	20
24	Pakowki.....	33.0	6,950	6,300	1475	1230	21
29	Cardiff.....	25.5	8,525	7,885	1505	1235	22
23	Magrath.....	9.8	10,740	10,215	1550	1275	23
15	Lundbreck....	6.7	11,190	10,720	1560	1280	24
13	Sterling.....	7.3	9,730	9,260	1530	1280	25
26	Pembina.....	19.9	8,995	8,460	1515	1305	26
2	Luscar.....	3.7	12,510	12,055	1585	1320	27
36	Big Valley...	20.9	8,375	7,830	1500	1355	28
4	Brazeau.....	1.8	13,505	13,080	1600	1380	29
6	Hillcrest....	1.3	12,200	11,780	1575	1525	30
1	Blue Diamond.	1.3	12,585	12,180	1585	1550+	31
7	Greenhill....	2.1	12,915	12,490	1590	1550+	32
3	Cadomin.....	2.4	12,640	12,200	1585	1550+	33

The values for the moisture content and the heating value are the average of the values obtained in the last two years in the laboratory of the Industrial Research Council.

The Clinkering Index

From the figures obtained as described above, those representing the softening temperature of the coal ash were subtracted, giving the difference in degrees Centigrade, and it is suggested that these be used as an index of the clinkering tendency of the coals. It is probable that the figure so obtained should be modified to some extent by the amount of ash in the coal, for there will be a distinct loss of heat due to the temperature at which the ash is removed from the fuel bed, and also a slight loss of heat due to fusion, if any has taken place. The relation does not depend on the amount of the ash directly, as pointed out above, unless it has little or no low melting constituents. When this is the case, it is likely that with a small index and a large amount of ash, little or no clinker will form.

A table has been prepared, showing the clinkering index of the samples, together with the typical ash associated with them, but information is not at present available as to the low melting constituents of the ash. The flowing interval is also indicated in Table XII.

The order of the same samples, when listed with regard to the softening temperature of their ash is also indicated, and it will be seen that, for the most part, they are not notably displaced. This method of estimation offers a solution for the problem of why the amount of clinker formed is not

TABLE XIII.- ASH SAMPLES IN ORDER OF THEIR CLINKERING INDEX,
SHOWING ALSO FLOWING INTERVAL, PERCENTAGE OF ASH IN COAL,
AND ORDER WHEN ARRANGED ACCORDING TO SOFTENING TEMPERATURE

No.	Mine	Clinkering Index	Flowing Interval	Ash, %	Order of Softening Temperature, as in Table XI.
37	Wayne.....	495	5	6.4	1
38	Drumheller.....	465	55	5.3	2
41	Three Hills.....	455	55	9.0	3
14	Saunders.....	450	80	8.4	9
5	Canmore.....	450	115	7.2	17
11	Mercoal.....	435	25	8.6	10
25	Redcliff.....	425	50	8.5	4
30	Tofield.....	420	25	5.0	5
39	Carbon.....	420	105	9.9	7
32	Can. Dinant.....	415	90	4.8	6
21	Galt.....	410	55	9.3	14
33	Spicer.....	395	20	4.6	8
34	Ardley.....	385	55	8.9	12
31	Dobell.....	375	20	4.5	11
40	Rumsey.....	365	100	13.1	13
27	Penn.....	360	85	6.5	15
35	Alix.....	360	55	7.7	16
38	Twin City.....	340	85	6.1	18
22	Coalhurst.....	340	55	9.8	20
12	Foothills.....	340	40	11.9	19
15	Lundbreck.....	280	60	16.8	24
23	Magrath.....	275	55	12.5	23
29	Cardiff.....	270	25	6.7	22
2	Luscar.....	265	100	16.1	27
13	Sterling.....	250	65	20.3	25
24	Pakowki.....	245	35	8.2	21
4	Brazeau.....	220	60	14.5	29
26	Pembina.....	210	20	9.6	26
36	Big Valley.....	145	25	11.8	28
6	Hillcrest.....	50	17.4	30
3	Cadomin.....	40-	14.9	33
7	Greenhill.....	35-	13.8	32
1	Blue Diamond..	35-	16.7	31

always in accord with the softening temperature. E.G.Bailey, quoted above, mentions this trouble, and so does Ricketts in his report to the meeting of the A.S.T.M. in 1923. (See Proceedings of the A.S.T.M. for that year.)

It should be noted that the clinkering index, as well as the values for softening temperature, would indicate that no clinkering would take place with a number of the high ash bituminous coals, whereas in each case a certain amount of clinker is formed. An explanation of this discrepancy, based on the fact that different portions of a particular ash fuse at different temperatures, was outlined above under "Constitution of the Ash."

Observations From Boiler Trials

To obtain first hand information of the clinker trouble given by all the representative Alberta coals in actual practice would be an almost impossible task. To obtain comparative results, the coals should be burned in the type of furnace most suited to their particular qualities; the best type for the high volatile coals of the prairies is not the best for the bituminous coals of the mountains; and so to test them all, a series of different installations would be necessary.

The amount of ash in any one kind of coal varies from time to time, and a series of runs would have to be made to get average results; for the amount of ash will in certain cases influence the amount of clinker to a different degree, depending

on the uniformity of the constituents.

The degree to which the fire is forced will be an important factor, owing to the higher temperature produced at higher ratings, so that it is difficult to make comparisons from this point of view.

The heating value of the coals as mined at present is slightly above the values recorded in Bulletin 25, published in 1918. The difference may be due to methods of mining, sampling, or calorimetry, or to an increase in the heating value of the coal. If this last is true, the difference is probably greater when applied to results obtained some 10 or more years ago. The general average of other characteristics may have changed too, and the clinker formation given by a coal some time ago will not therefore necessarily be duplicated by present day samples from the same mine.

Boiler trials have been run on different occasions, and under different circumstances, by different authorities; and though it is realized that they are not strictly comparative for the reasons given above, they have been assembled in one list for convenience. The observers are given in every case, and, generally, comparisons should be limited to the coals quoted by any one authority. In the case of coals used in boiler trials in the power plant of the University of Alberta and at other institutions in this city, and credited variously to R.T.H., McM, A.G.S., and *A.G.S., the estimation of the degree of clinker is comparative, and though most of these trials were

conducted in furnaces designed especially for lignite coals, some of the bituminous and sub-bituminous trials were made on a marine type, so that these, together, make the nearest approach so far obtained to a comparative measure of the clinkering tendency of a series of Alberta coals. The results to which these remarks apply have been starred (*) in the list below,

Table XIII.

With regard to one of these results (Sterling, A.G.S.) in which practically no clinker was formed, it may be stated that the softening temperature of the ash from this sample of coal was determined, and was more than 100°C. above the average sample, so that the index for this trial was actually less than zero.

Comparison of Table XII., showing the clinkering index of the various samples, with the comments on clinker formation in practice in Table XIII. shows that the general tendency is very well indicated. In general it may be said that coals with an index below 300 will give little trouble from clinker, but that low ash coals with an index above 300 will form clinker, giving more or less trouble; and that in comparing coals with approximately the same index and amount of ash, the one with the shortest flowing interval will form the most clinker.

TABLE XIII.- OBSERVATIONS ON CLINKER FORMING PROPERTIES OF
ALBERTA COALS DURING THE PROGRESS OF BOILER TRIALS,
Coals arranged in order of their Clinkering Index.

Coal	Remarks	Observer
Drumheller.....(1)	Large amount of clinker over bars. Cleaned six times in 11 hours.....	J.B.
" (2)	Very little trouble, easily removed..	J.B.
" (3)	Clinker in large slabs over grates...	J.B.
" (4)	Thin clinker, spreads on bars and sticks a little.....	J.B.
* "	Large amount of clinker formed.....	*A.G.S.
*Saunders.....	Large slabs of clinker spread over grate, easily removed (2 trials)...	A.G.S.
Canmore.....	Clinker hard, some trouble.....	R.J.D.
" washed.....	Clinker hard, sticking to bars without steam.....	R.J.D.
Bankhead.....	Large pieces of clinker, sticks without steam.....	J.B.
"	Large pieces, very soft, easily removed	J.B.
Mercoal (Coalspur)	Clinker spreads over bars, easily removed.....	J.B.
*Tofield	Large slabs over the grate at high rating, not difficult to remove....	A.G.S.
Galt.....	Very viscous, improves with steam....	R.J.D.
* "	Somewhat worse than Edmonton.....	McM.
*Dobell.....	Small amount of clinker.....	R.T.H.
Penn.....	Clinker difficult without steam.....	R.J.D.
* "	Considerable difficulty at high rating	McM.
* "	Much soft clinker at all times.....	A.G.S.
Twin City.....	Clinker in hard small lumps, sticks slightly.....	J.B.
* " "	Small amount of clinker formed.....	R.T.H.
* " "	Better than Penn.....	McM.
*Black Diamond.....	Large amount of soft clinker.....	*A.G.S.
*Foothills.....	Very little clinker.....	McM.

(Continued on next page)

Coal	Remarks	Observer
Cardiff.....	Hard clinker forms in fairly large pieces does not stick.....	J.B.
* "	A small amount of clinker formed.....	*A.G.S.
Luscar.....	(See Mountain Park, below, under Cadomin)	
*Sterling.....	A few small pieces of clinker.....	A.G.S.
*(Coal Valley)...	A few small pieces of clinker formed.....	R.T.H.
*Pembina..... "	A small amount of clinker formed..... Clinker in small pieces, easily removed..	*A.G.S. J.B.
*Big Valley.....	Small amount of clinker formed.....	R.T.H.
Hillcrest..... " washed "	A small amount of hard clinker..... A small amount of hard clinker..... Clinker in small hard pieces.....	R.J.D. R.J.D. J.B.
*Cadomin..... * "	A few pieces of hard clinker..... A small amount of clinker, hard and soft	A.G.S. R.T.H.
Greenhill.....	A few small pieces of hard clinker.....	J.B.
*Blue Diamond...	A fair amount of large soft clinker.....	R.T.H.
(Jasper Park)..	Clinker in small pieces.....	J.B.

R.J.D. Durley, "Investigation of the Coals of Canada", 1910.

J.B. Blizzard Bulletin 27, Mines Branch, Ottawa, 1920.
" 331, " " "

R.T.H. Hollies Research Council of Alberta Boiler Trials, 1921-2

McM. McMillan Supt. Power Plant, University of Alberta, 1921-2

A.G.S. Scroggie Boiler trials personally observed, 1923-4

*A.G.S. " Information from firemen and engineers

CHAPTER VII.- EFFECT OF WASHING A COAL ON THE SOFTENING TEMPERATURE OF ITS ASH

Dr. J. B. Porter, in "An Investigation of the Coals of Canada, Vol.I.", deals very thoroughly with the subject of washing coals, and draws attention to the fact that as the better seams of an area are worked out, and as the consumers become more discriminating in their choice of fuel, coal washing is generally resorted to. He even says, on page 193, speaking of the introduction of coal washing plants: "The change will probably first be made in the coke-producing districts of the west."

He lists as advantages to be gained by washing:-

- (1) Improvement of the heating value;
- (2) Saving of transportation and labor costs in handling inert matter;
- (3) Improvement of the quality of the coke;
- (4) Removal of pyrite lessens the tendency to clinker;
- (5) Lowering of the sulfur and phosphorus content of the coke.

This is a most important matter if the coke is to be used for the production of iron and steel, in which these elements must be reduced to the last trace. This is becoming more and more significant as the quality of the iron ore mined becomes poorer.

Figures obtained in the analysis of coal ash have been used to estimate the amount of sulphur and phosphorus in the coke from different bituminous coals, but this method takes no account of

the amount of organically combined sulphur which is lost in preparing the ash, but is not lost during the coking.

TABLE XIV.- SULPHUR AND PHOSPHORUS IN THE ASH AND COKE OF SOME ALBERTA BITUMINOUS COALS.

Mine	Ash in coal, %	S O ₃		P ₂ O ₅	
		In ash, %	In coke, %	In ash, %	In coke, %
Brule.....	16.7	.53	.1	.74	.14
Nordegg.....	14.5	3.57	.62	.44	.08
Canmore.....	7.2	.07	.006	8.18	.68
Greenhill....	13.8	.07	.01	.63	.11
Hillcrest....	17.4	.80	.19	.14	.04
Limits.....	10.0	1.5002 to .05

The limits given in each case are taken from Illingworth, "The Analysis of Coal & Its Byproducts," and they show that to produce first quality coke most of our bituminous coals will have to be washed. These figures, as suggested above, represent a minimum of phosphorus and sulphur, and it is likely that more of these elements are left in the coke, as it is made, than is given from these figures derived from the coal ash.

Coal washing on a large scale is not practicable, but a sample of Greenhill coal was washed by Mr. Cameron with the washing apparatus available in the Mining Department. Two other samples available were tested by means of heavy solutions having a gravity of 1.55. This is recommended by Porter as giving a good indication of the probable success and limits of actual

washing. The results of these tests were as follows:-

TABLE XV.- RESULTS OF WASHING AND HEAVY SOLUTION SEPARATIONS

Sample	Recovery	Ash in Coal, %	Ash after Washing, %
Greenhill.....	73.0%	17.4%	12.2%
Luscar.....	83.8	16.1	7.8
Sterling.....	77.2	18.8	10.2

* The softening temperature of the ash from the feed, waste, and recovery, was run in each case.

TABLE XVI.- FUSION DATA IN RELATION TO ASH CONTENT

No.	Co a l	Softening Temp.	Softening Interval	Flowing Interval	Ash, %
52	Greenhill, Feed.	1550+°C			
51	" Washed	1550+°C	The washed sample appeared to		
53	" Waste..	1550+	be slightly softer.		
15	Luscar, Feed....	1320	210°C.	100°C.	16.1
70	" Float...	1360	260	80	7.8
71	" Waste...	1350	180	90	
82	Sterling, Feed..	1390	240	75	18.8
80	" Float.	1385	215	60	10.2
81	" Waste.	1335	140	70	
82	Sterling, com- mercial.....	1390	240	75	18.8
13	", inspector.	1280	170	65	20.3
41	Three Hills.....	1075	75	55	6.1
41A	" "	1320	230	60	13.5

SUMMARY OF ABOVE RESULTS

	Sample	Ash Reduction	Softening Temp. Change	Softening Interval Change
	Greenhill.....		No conclusions owing to infusibility	
	Luscar.....	16.1-7.8	Raised 40°C.	Raised 50°C.
	Sterling.....	18.8-10.2	Lowered 5°C.	Lowered 35°C.
	Sterling.....	20.3-18.8	Raised 110°C.	Raised 70°C.
	Three Hills.....	13.5-6.1	Lowered 245°C.	Lowered 155°C.

The last two examples are distinct samples, and not washed. The flowing interval shows little difference in any case. These erratic results have been found by other investigators.

In a report on the Trent Process for the removal of mineral matter from coal (Bureau of Mines, Report of Investigation, Serial No. 2263), the following changes resulted from the reduction of ash content.

TABLE XVII.- RELATION OF FUSION DATA TO ASH REDUCTION, TRENT PROCESS

Sample	Ash Reduction	Softening Temp. Change	Softening Interval Change
Freeport	9 to 7	Same	Increased 260°C.
Pittsburg.....	12 to 6	"	" 160
Pittsburg.....	12 to 5	Lowered 30°C.	" 170
Indiana.....	9 to 6	Increased 170	" 210
Oklahoma.....	18 to 8	" 20	Lowered 120

It will be noted that the softening temperature was raised, lowered, and unaltered, in different cases.

Stansfield, working at McGill several years ago, fused the ash of several coals before and after washing. (Investigation of the Coals of Canada, Vol. 2.) His results are not comparable with those given above, since a different type of furnace was used, and different methods of fusion measurement. He concluded that washed coal gave an ash less fusible than the ash from the same coal when unwashed. I have endeavored to interpret his results on a comparative basis.

TABLE XVIII.- FUSION DATA IN RELATION TO ASH CONTENT, STANSFIELD

Sample	Ash Reduction	Softening Temp. Change	Softening Interval Change
Bankhead.....	12.2 to 8.9	Increased 100+	
Canmore.....	12.3 to 5.9	Both infusible at 1500°C., washed sample appears slightly softer.	
Glace Bay.....	5.9 to 2.7	Interval increased

These results are more or less fragmentary, but suggest that no definite conclusion can be drawn with regard to the effect that washing a coal will have on the fusibility of its ash. These practical results are in accord with the statement in Bulletin 129, page 14, which is apparently based on theoretical grounds alone. It says, "Washing may either increase or decrease the ash fusibility, depending on the fusibility of the intrinsic ash and the nature of extraneous impurities."

It should be noted that neither washing with water nor separation by means of the Trent Process works as well on iron pyrite as on other minerals, owing to the differential wetting of the mineral, and the presence of scaly particles. Results, therefore, obtained on pyritic coal will not be strictly comparable with those from pyrite-free coal.

The relative amounts of the different constituents of the extraneous ash, their different susceptibility to washing or separation, and their difference in softening temperatures are all factors that enter into the effect that washing will have on

a coal, so that no definite statement can be made. It may be said, in general, however, that where the mineral matter is principally of a clay shale or slate, a reduction might be expected; but if the mineral matter is formed of ankerite or pyrite, the washed coal will have an ash with an increased softening temperature; and when all are present in quantity, no prediction can be made.

CHAPTER VIII.- RELATION BETWEEN CHEMICAL COMPOSITION AND SOFTENING TEMPERATURE.

Nature Of Fusion Of Ash

It is evident that coal ash, being a mixture of various constituents, will not have a definite melting point which is the criterion of a pure substance. The presence of each element will affect the others, so that the resulting softening is an accumulative effect, due to the number of different elements present, their relative amounts, and, in some cases, such as iron, their state of oxidation. In other cases, such as sulphur, (which may be present as organic matter, as sulphates, or as sulphides) the state of combination may make a difference in the effect on the softening temperature. It has already been suggested that some oxides occur free, and others in combination with silica, etc., so that this too may affect the influence of these elements, increasing the effect of some portions above other portions of the same element, or, possibly, in the case of free oxides, removing them from the reaction altogether.

In general, when two oxides are fused together, the melting point of the mixture is lower than that of either oxide separately, and there is a certain mixture of the two that gives the lowest possible melting point. These are known as the eutectic mixture and eutectic temperature. Given such a mixture, the addition of more of either substance raises the fusing temperature

so that the addition of an oxide to another oxide may first lower the fusing temperature; and, later, if the amount added is increased, may raise the temperature. As examples of the lowering of fusing temperatures, Washburn, "Principles of Physical Chemistry", gives the following:-

CaO, fusing at 2570°C., and SiO₂, at 1625, form an eutectic which melts at 1436°C.

CaO, fusing at 2570°C., and Al₂O₃ at 2050°C., form an eutectic melting at 1400°C.

The addition of a third oxide causes a further lowering, as in the lime-silica-alumina system an eutectic melting at 1265°C. is formed. Addition of a fourth and a fifth component, if in small amounts, causes still further lowering. In ordinary samples of coal ash there are some ten or eleven different elements, so that the involved nature of the fusion reactions is at once apparent. The effects produced by one oxide on the other is in proportion to its molecular concentration, so that any attempt to solve the fusion temperature by means of the analysis of the ash should presumably start from that point.

Certain of the constituents are present in small amounts. These are, as a general rule, phosphorus, sulphur, manganese, magnesium, titanium, and the alkalis; while the bulk of the ash is made up of the oxides of silica, alumina, calcium, and iron. These latter usually amount to 75% of the weight of the ash. Fieldner gives as typical limits the following:-

Silica.....40% to 60%
 Alumina....20% to 25%
 Ferric Oxide, 5% to 25%

Lime..... 1% to 15%
 Magnesia...0.5% to 4%
 Alkalies... 1% to 4%

The results of the chemical analyses made in this investigation are given in Table XIX., where the percentage composition of the different ash samples, arranged in order of their softening temperatures, is tabulated.

TABLE XIX.- PERCENTAGE COMPOSITION OF ASH SAMPLES,(ELEMENTS CALCULATED AS OXIDES) ARRANGED IN ORDER OF SOFTENING TEMPERATURE

No.	Soft- ening Temp. °C.	Sam- ple No.	Oxides , percent								
			SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O K ₂ O	SO ₃	P ₂ O ₅	TiO ₂
1	1085	32	36.35	21.85	7.60	19.30	1.64	5.45	5.99	1.26	1.00
2	1140	21	49.20	18.59	5.22	13.81	1.59	4.04	3.78	3.05	1.30
3	1150	27	55.20	21.70	5.00	9.88	1.13	4.45	1.88	.46	.90
4	1170	28	46.82	21.58	3.98	18.80	1.23	5.28	.88	.56	.90
5	1170	5	36.10	31.67	14.54	2.83	1.64	4.31	.07	8.18	1.44
6*	1210	12	42.08	12.95	4.32	32.40	1.67	**	1.87	.22	.72
7	1210	22	45.70	24.15	7.60	12.73	1.93	3.68	3.47	.35	.95
8	1280	13	59.80	23.76	3.03	8.00	.99	2.17	1.62	.32	1.20
9	1355	36	52.30	27.58	2.83	11.78	.99	3.58	.08	.03	1.20
10	1380	4	53.10	26.44	5.23	6.22	1.93	2.21	3.57	.44	1.60
11	1525	6	55.30	32.83	5.45	2.94	.59	.56	.80	.14	2.00
12	1550	7	58.97	27.18	6.00	1.93	1.34	**	.07	.62	2.50
13	1550	1	59.15	28.61	3.59	3.45	1.20	.44	.53	.74	2.10

* This ash contains a small amount of MnO.

** Alkali not determined.

Table XX. shows the same results, but in this table the elements are shown in molecular proportions. It is apparent that in only occasional cases is there enough phosphorus present to make any material difference in the softening temperature. It should be determined, however, unless some method for isolating alumina

separate from phosphorus is used in the analysis. The percentage of phosphorus present, if it is not deducted from the alumina, may make a large error. Titanium, in a similar fashion, unless isolated, may appear as part of the aluminium or iron oxides, depending on the methods of analysis used.

TABLE XX.- MOLECULAR PROPORTIONS OF OXIDES, SAMPLES ARRANGED
IN ORDER OF THEIR SOFTENING TEMPERATURE.

Order of Softening Temperature	Sample No.	Molecular Proportions = $\frac{\text{percentage} \times 100}{\text{molecular wt. of oxide}}$								
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ K ₂ O	SO ₃	P ₂ O ₅	TiO ₂
1 ...	32	606.	212.	47.5	345.	40.7	64.6	74.9	.88	12.5
2 ...	21	816.	182.	32.7	247.	39.4	50.7	47.3	2.15	16.2
3 ...	27	917.	213.	31.3	176.	28.0	69.2	23.5	.32	11.2
4 ...	28	777.	211.	24.9	336.	30.4	61.4	11.0	.39	11.2
5 ...	5	598.	310.	91.0	50.5	40.7	49.5	7.9	5.75	18.0
6 ...	12	696.	126.	27.1	578.	41.3	**	23.4	.15	8.9
7 ...	22	774.	236.	47.5	227.	47.8	41.6	43.4	.24	11.8
8 ...	13	992.	232.	19.0	143.	24.5	29.6	20.3	.22	15.0
9 ...	36	867.	270.	17.7	210.	24.5	51.3	1.0	.02	15.0
10 ...	4	881.	259.	32.8	111.	47.8	24.6	44.6	.31	20.0
11 ...	6	917.	322.	34.1	52.5	14.6	8.1	10.0	.09	25.0
12 ...	7	977.	265.	37.5	34.5	33.2	**	7.9	.44	31.2
13 ...	1	980.	281.	22.5	61.6	29.7	5.8	6.6	.52	26.3

** Alkali not determined

In connection with the occurrence of phosphorus in Alberta, Dr. Allan draws attention (4th Annual Report on the Mineral Resources of Alberta, 1922, p.65) to the fact that there are phosphate-bearing rocks in the series just under the Kootenay in which the bituminous coals are found. A small vein has been located near Banff, and in the Canmore coal sample from near Banff, too, there is an unusually high phosphorus content. If there is any connection between these facts, it would indi-

cate that the most likely place to find phosphates in quantity in Alberta would be in this vicinity.

TABLE XXI.- RELATION OF PERCENTAGE COMPOSITION (OXIDES)
OF THE ASH SAMPLE TO THE SOFTENING TEMPERATURE

Serial Sample No.	Softening Order, Table	Order of Oxide Content						
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O K ₂	TiO ₂	SO ₃
32 ...	1	2	5	12	12	11	5	13
21 ...	2	6	2	7	10	7	8	12
27 ...	3	9	4	6	7	9	3	9
28 ...	4	5	3	4	11	10	2	6
5 ...	5	1	12	13	2	8	9	2
12 ...	6	3	1	5	13	**	1	8
22 ...	7	4	7	11	9	6	4	10
13 ...	8	13	6	2	6	3	6	7
36 ...	9	7	10	1	8	5	7	3
4 ...	10	8	13	8	5	4	10	11
6 ...	11	10	8	9	3	2	11	5
7 ...	12	11	9	10	1	**	13	1
1 ...	13	12	11	3	4	1	12	4

** Alkali not determined.

Relation Between Chemical Analysis And Softening Temperature

From the work which was discussed above under "Constitution of the Ash," it would be expected that silica and alumina would act as refractory components, and that calcium, iron, and magnesium would tend to lower the softening temperature, particularly if the iron were present in the ferrous state, which is normal for ankerite residues. The presence of alkali will also lower the softening temperature, but as a rule neither sodium nor potassium is present in any large amount.

Examination of Tables XIX. and XX. shows that these elements as a general rule had the effect that would be expected, but that the influence of one is often masked by the influence of the others, so that there is no regular relation between the softening temperature and the percentage composition of any one oxide. The table, however, shows that there is generally, among the samples with high softening temperatures, a larger proportion of silica, alumina, and titanium, while in those that soften at the lower temperatures there is an increase in the amount of calcium, magnesium, iron, sulphur, and the alkalis.

out

These relations are brought/more clearly in Table XXI., where the samples are arranged in order of increasing softening temperature. In the columns for each oxide the numbers show the order of increasing percentage of that oxide. Thus, under silica, sample no.1 has the second lowest silica content, and

sample no. 5 the lowest. The lack of order in these figures, due to the various influences stated above, show what a difficult problem it is to find a relation between the chemical analysis and the softening temperature.

Various workers have at different times attempted to derive formulae that would show the relation between the chemical analyses and the softening temperatures. As a rule these work successfully with some, but not with others. Examples are:-

(1) Bischoff's formulae for a fire clay,-

$$\frac{\text{Oxygen in Alumina}}{\text{Oxygen in Fluxes}} \div \frac{\text{Oxygen in Silica}}{\text{Oxygen in Alumina}}$$

gives a quotient which rises with increasing softening temperature, and which is stated by Prost to apply to coal ash. If the quotient is above 3, the ash will not soften below 1500°C.; while if the quotient falls to 1, the ash will be fusible, and melt below 1300°C.

(2) Dr. J. T. Dunnin (J. Soc. Chem. Ind., Vol.37, p.15.) lists the results of the chemical analysis and the softening temperatures of samples of ash that he had prepared. He suggests, as a guide, that where the ratio,-

Molecular proportions of Alumina to 1,000 molecules Silica
Molecular proportions of Other Bases to 1,000 molecules Silica

exceeds 1.75, there will be little trouble from clinkering.

From the relations brought out in the work done in this investigation, it is suggested that the ratio,-

Sum of molecular proportions of SiO₂, Al₂O₃ and TiO₂
Sum of molecular proportions of CaO, MgO, Fe₂O₃, Alkalies & SO₃

gives a quotient which is above 10 for infusible ash samples, and it is therefore an indication of probable clinkering tendencies.

It is to be noted that this formula is formed on a purely empirical basis, there being both bases and acid radicals in numerator and denominator. Further analyses might cause a change in this formula, for it is drawn from the apparent relation between chemical composition and softening temperature which is shown by the thirteen samples analyzed. Table XXII. gives the results of the application of these formulae to the samples analyzed.

TABLE XXII.- APPLICATION OF FORMULAE RELATING CHEMICAL ANALYSIS AND FUSIBILITY OF ASH, SAMPLES IN ORDER OF THEIR SOFTENING TEMPERATURE

Serial No.	Softening Temperature	Bischoff	Dunn	Scroggie
32	1085°C.	0.60	0.4	1.4
21	1140	0.44	0.5	2.4
27	1150	0.65	0.7	3.4
28	1170	0.53	0.5	2.1
5	1170	1.80	1.3	3.9
12	1210	1.52	0.2	1.2
22	1210	0.75	0.6	2.5
13	1280	1.02	1.1	5.2
36	1355	1.18	0.9	3.8
4	1380	1.27	1.2	4.4
6	1525	2.87	2.9	10.6
7	1550	1.81	2.5	11.3
1	1550	2.27	2.3	10.2

It will be noted that neither of the three ratios gives a quotient which varies directly with the softening temperature. This is due, presumably, in part at least, to the effect of oxidation and combination as outlined above.

The claims made for Bischoff's formulae are not upheld for ash samples nos. 6, 7, and 1, none of which show a quotient **above 3**, and soften below 1500°C.; while **three of the samples** softening below 1300°C. **have a quotient above 1.**

Dr. Dunn's formula shows no exceptions. The samples with a quotient above 1.75 are all infusible at 1500°C., and give no trouble with clinkers.

The formula suggested by this work gives a quotient above 10 for an infusible ash, and is probably no better than the one suggested by Dr. Dunn.

Burgwin, H.K., jr., (J.Ind. & Chem.Eng., Vol.6,(1914), p.694.) concludes that where Fe_2O_3 is less than 10%, the fusing temperature is not below 1399°C. (All the samples in Table XIX. are contrary to this statement); where the Fe_2O_3 in ash is more than 20%, the temperature is never above 1399°C. (In Table XIX. none of the coals have this amount of iron.); that where S and Fe_2O_3 in coal is below 3%, the fusing temperature is above 1399°C. (Most of the samples are contrary to this also; e.g., no.32 contains Fe_2O_3 , 7.6%, and S, 2.4%, in approximately 5% ash coal, so that the content of Fe_2O_3 and S is about 0.5% in the coal, and the ash softens at 1085°C.); that where S and Fe_2O_3 is above 3% in

the coal the ash will fuse below 1399°C. (None of the samples in Table XIX. fall in this class.). It is to be noted that the figure 1399°C. might not be duplicated in the standard method of determining the softening temperature, but the exceptions noted above could hardly be accounted for on this basis.

In considering the relation between chemical composition and softening temperature it is interesting to note that only one of the samples has an excess of acidic equivalents, and this one, no. 13, softens at 1280°C., almost half way between the lowest and highest softening temperatures obtained in this series of tests.

Relation Between Chemical Analysis & Intervals

Reference to the flowing intervals, as shown in Figure No.3, reveals that they vary without any apparent regularity. The softening intervals, on the other hand, shown in the same figure, are fairly consistent: the average being about 50 to 60°C. for all ash samples having a softening temperature below 1165°C.. For those above this temperature, beginning with 1170°C., the softening interval is much longer, being about 150°C. for those whose softening temperatures lie between 1170° and 1270°C.; and for those whose softening temperatures are above 1270°C., the average is about 200°C.. Notable exceptions to the groups are the sub-bituminous coals from the Coalspur area, viz., Mercoal and Foothills, the latter of which, on analysis, reveals a

large amount of calcium in its composition, so that this element may play an important part in the viscosity of the clinker. It apparently causes a more fluid clinker, while iron oxide, alumina, and silica are highly viscous, as shown by the small amount of bending in the highly siliceous ash of the bituminous coals, and the viscosity of the ankerite residues ("Constitution of the Ash").

Some Recent Opinions On The Relation Of Chemical Analysis To Softening Temperature

Mr. Sinatt (in his report of the Lancashire & Cheshire Coal Research Association, 1920) concludes "that chemical analysis is of little value as a criterion of the melting point of ash." Dr. R. Lessing, in commenting on this, said; "There is no question that Richter's Law, according to which fluxes act on clays and other refractory materials in molecular ratio, still holds good, and that melting points can be calculated from the analytical results, provided the analysis is complete in all its details, including the state of oxidation and the manner of combination of the various constituents." Mr. Sinatt, in his reply, pointed out that such knowledge was at present impossible to obtain completely, owing to the heterogeneous nature of coal ash.

Bone, in his monograph, "Coal and Its Scientific Uses," (1919), says that the relation of fusibility of the ash of a coal to its chemical composition is a very important, though

somewhat complex question, concerning which the present state of our knowledge is not sufficiently definite to warrant the laying down of any precise rules.

Dr. Dunn (1918), whose work is quoted above, concluded that although at present no law can be formulated, the melting point as a rule rises as the relative molecular proportions of basic oxides to silica diminishes. (Bone, pg.41.)

At the commencement of this investigation it was hoped to find out rather more about the relation between the softening temperature and the chemical composition than can be reported. The writer, however, hopes that the analyses of these samples of ash, whose softening temperatures has also been determined, will prove of use in some future investigation.

Effect Of The Addition Of Foreign Matter On The Softening Temperature Of The Ash

It had been intended to do some work with the low-fusing coals, in order to determine whether the addition of some common substances, such as sand, limestone, or clay, would alter the softening temperature sufficiently to be worth practising on a commercial scale.

Lack of time prevented this being done, but from the results of the work under "Constitution of Ash" it can be said that the addition of fine sand/to samples already high in silica and alumina should raise the softening temperature of the ash as

determined in the standard method. It is very doubtful, however, if it would alter the amount of clinker formed to any appreciable extent. In the high ash bituminous coals studied it was shown that the standard method indicated that the ash was infusible at 1550°C., and yet a certain amount of clinker formed, which was presumably from the low-fusing compounds present. It seems probable, therefore, that the addition of sand or clay, which would behave very much like the extraneous ash in the samples referred to above, would not materially affect the amount of clinker formed.

The addition of limestone, on the other hand, which would form on ignition free calcium oxide, would tend to lower the softening temperature, since most samples of ash have an excess of silica over other oxides; and it would therefore act as a flux and lower the softening temperature until the amount added became so much that the total amount of lime was greater than the equivalent amount of silica, when further additions would tend to raise the softening temperature again, since the lowering effects of the other oxides would be diluted, and this action would depend directly on the amount of calcium already in the coal. The effect of these additions becomes directly linked up with the chemical analysis of the ash, and unless this is known, the effect of adding foreign material can not be intelligently studied.

L. S. Marks did some work along this line in 1910, but

his investigations were made before the standard method was evolved, and his results are therefore not strictly comparative with those obtained in the present investigation. He did not define the softening temperature, and so the degree of modification in it is not definite. He worked on a non-clinkering coal from West Virginia, whose ash had a softening temperature of 1400°C. He used cones in estimating the softening temperature, which he measured with a thermo-couple. His results are tabulated below.

TABLE XXIII.- EFFECT OF ADDITIONS OF FOREIGN SUBSTANCE ON THE SOFTENING TEMPERATURE OF ASH (L. S. Marks)*

Substance Added	Effect on Softening Temperature of Adding...		
	25%	50%	75%
Al ₂ O ₃	Raised	Raised	Raised
CaO.....	Lowered considerably	Raised
Fe ₂ O ₃	Lowered slightly	Lowered slightly	Lowered slightly
FeS ₂	Lowered considerably	Lowered considerably	Lowered considerably
CaS ₂	Lowered	"	"
CaSO ₄	"	"	"

* Engineering News, Vol.64 (1910), p.623-626.

No report is made of the effect of adding silica, nor is the chemical analysis of the sample given, but from the fact that it was a non-clinkering variety, it presumably had a high silica content.

These results are quite in line with what would be expected from the theoretical considerations given above, and suggest that some further work might give the proportion of lime to be added to raise the softening temperature; or, in general, the amount of foreign substance that would have to be added to raise the softening temperature sufficiently to prevent the fusion of much of the ash. It seems highly desirable that this work should be done on samples which have been analyzed chemically.

Whether this would work in practice or not is a matter that could only be determined by actual work under furnace conditions, but the amount of matter to be added could be determined to some extent by the use of cones of the original ash, and of others to which different proportions of foreign matter had been added.

Methods Used in Chemical Analysis

Silica	-- True Silica	-- Scott, 1922, page 436.
Iron	-- Volumetric	-- Treadwell & Hall, 1919, pg.109.
Aluminium	-- Gravimetric	-- ditto , page 111.
Titanium	-- Method of Weller	-- ditto , page 100.
Calcium	-- Volumetric	-- Scott , 1922 , page 108.
Magnesium	-- Gravimetric	-- ditto , page 293.
Sulphur	-- Gravimetric	-- ditto , page 497
Phosphorus	-- Gravimetric	-- J.Ind.& Eng.Chem., <u>15</u> ,44
Sodium)	-- J.Lawrence Smith Potassium)	-- Scott, 1922, pages 416 method and 413.

S U M M A R Y

1. Thirteen samples of ash have been analysed, and their softening temperatures determined.
2. The softening temperatures of twenty other ash samples have been determined, the complete series of samples representing all the typical Alberta coals.
3. A method has been suggested for taking into account the heat of the furnace in determining the tendency to clinker.
4. A method has been suggested for estimating the net calories of a coal without the use of its ultimate analysis.
5. The general results obtained by the A.S.T.M. standard method for determining the softening temperature of coal ash have been compared with clinker formation in practice. Explanations have been offered for cases where the standard method fails to indicate clearly the tendency to clinker.
6. The relationship of the constitution of the ash to the softening temperature has been discussed, and the different components of two ash samples tabulated.
7. The effect on the softening temperature of reducing the ash of a coal by washing, heavy solution separation, or oil separation has been shown to vary with the sample, and the reason for this is pointed out.
8. The chemical analyses of ash samples have been compared with their softening temperatures, but no definite relation has been secured.

BIBLIOGRAPHY

A very complete bibliography is given in Bulletin 129 of the U.S. Bureau of Mines, Washington, (1918), classified under the following headings:-

Fusibility and Clinkering of Coal;
Contributions to the Physical Chemistry of Slag & Silicates;
Melting and softening temperatures of oxides, sulphides,
silicates, mixtures, and slags;
Pyrometric Cones, their use in measuring temperature;
Pyrometers, and the measurement of high temperature.

Some more recent contributions to the subject have been used, and others not included in the above list are referred to in the text. These include:-

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